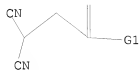


10/522,764

=> d  
L1 HAS NO ANSWERS  
L1 STR



G1 O,S

Structure attributes must be viewed using STN Express query preparation.

=> s l1 full  
REGISTRY INITIATED  
Substance data SEARCH and crossover from CAS REGISTRY in progress...  
Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

FULL SEARCH INITIATED 15:53:36 FILE 'REGISTRY'  
FULL SCREEN SEARCH COMPLETED - 1160 TO ITERATE

100.0% PROCESSED 1160 ITERATIONS 208 ANSWERS  
SEARCH TIME: 00.00.01

L2 208 SEA SSS FUL L1

L3 78 L2

=> s l3 and py<2002  
21939583 PY<2002  
L4 68 L3 AND PY<2002

=> s l4 and halo?  
407321 HALO?  
L5 4 L4 AND HALO?

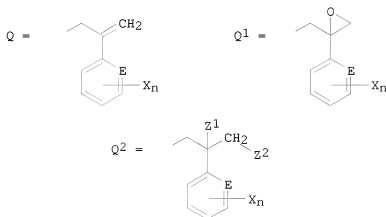
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L5 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2008 ACS on STN  
ACCESSION NUMBER: 1998:76233 CAPLUS  
DOCUMENT NUMBER: 128:177233  
TITLE: Malononitrile derivatives and herbicides containing them  
INVENTOR(S): Hosokawa, Akemi; Ikeda, Osamu  
PATENT ASSIGNEE(S): Mitsubishi Chemical Industries Ltd., Japan  
SOURCE: Jpn. Kokai Tokkyo Koho, 18 pp.  
CODEN: JKXXAF

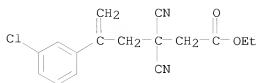
DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 10029966	A	19980203	JP 1996-187796	19960717 <--
PRIORITY APPLN. INFO.: OTHER SOURCE(S):	MARPAT 128:177233			19960717

GI

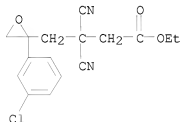


- AB The derivs. are represented by R1R2C(CN)2 [I; R1 = H, C1-6 alkyl, C4-7 cycloalkyl, C2-6 alkenyl, C2-6 alkynyl, C1-6 haloalkyl, C2-6 haloalkenyl, C2-6 haloalkynyl, C2-8 alkoxyalkyl, C3-6 alkoxyalkenyl, C2-4 cyanoalkyl, C1-6 hydroxyalkyl, C2-7 alkylamido, C7-9 aralkyl, C8-12 arylcarbonylalkyl, (un)substituted Ph, (un)substituted pyridyl, (un)substituted thiazolyl, CR3R4A; A = (un)substituted Ph, pyridyl, thiazolyl; R3-4 = H, Me; R2 = Q, Q1, Q2; E = CH, N; X = halo, C1-4 alkyl, C1-3 haloalkyl, NO2, C1-8 haloalkoxy, (un)substituted benzyloxy, pyridyloxy; n = 0-2; Z1-2 = OH, halo, C1-4 alkylsulfonyloxy, (un)substituted phenylsulfonyloxy]. The herbicides contain I as active ingredients. I (R1 = H, R2 = Q, E = CH, X = 3-Me, 5-Me) showed 91-100% herbicidal activity against Echinochloa oryzicola, Monochoria vaginalis, and Scirpus juncoides.
- IT 203127-60-0P 203127-94-0P  
 RL: AGR (Agricultural use); BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation); USES (Uses) (preparation of malononitrile derivs. as herbicides)
- RN 203127-60-0 CAPLUS
- CN Benzenepentanoic acid, 3-chloro-β,β-dicyano-8-methylene-, ethyl ester (CA INDEX NAME)



RN 203127-94-0 CAPLUS

CN Oxiranebutanoic acid, 2-(3-chlorophenyl)-β,β-dicyano-, ethyl ester (9CI) (CA INDEX NAME)



L5 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1993:213100 CAPLUS

DOCUMENT NUMBER: 118:213100

ORIGINAL REFERENCE NO.: 118:36739a, 36742a

TITLE: Preparation of tricyclic fused pyrimidine compounds

INVENTOR(S): Akimoto, Hiroshi; Otsu, Koichiro; Miwa, Tetsuo

PATENT ASSIGNEE(S): Takeda Chemical Industries, Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 25 pp.

CODEN: JKXXAF

DOCUMENT TYPE:

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 04211063	A	19920803	JP 1991-65613	19910305 <--
			JP 1990-54620	A1 19900305

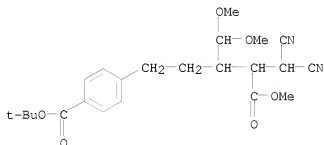
PRIORITY APPLN. INFO.:

OTHER SOURCE(S): MARPAT 118:213100

GI For diagram(s), see printed CA Issue.

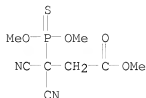
AB The title compds. [I; Q1 = H, halo, radical linked through C, N, O, or S; one of Q2 and Q3 = N, the other = N, CH; Y = N, CR1 (wherein R1 = H, hydrocarbonyl), methylidyne; Z = C2-5 bivalent radical containing optional substituents; ring A1, A2 = (substituted) 5-7-membered ring; B = (substituted) cyclic radical, etc.], useful as antitumor agents with high selectivity, are prepared by cyclocondensation of 1.181 g ester II (preparation given) with 314 mg guanidine HCl and Me3COK in Me3COH gave 1.02 g pyrrolopyrimidine III, which (1.010 g) was treated with borane-THF complex in THF at 0° and then at 50°, the solution cooled and stirred with HOAc-MeOH at room temperature to give 542 mg IV. The preferred doses of I are 2.0-500 mg/kg-day orally and 1.0-200 mg/kg injection.

IT	147239-87-0P
	RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
	(preparation and reaction of, in preparation of antitumor agent)
RN	147239-87-0 CAPLUS
CN	Benzenepentanoic acid, $\alpha$ -(dicyanomethyl)- $\beta$ -(dimethoxymethyl)-4-[(1,1-dimethylethoxy)carbonyl]-, methyl ester (CA INDEX NAME)



L5 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2008 ACS on STN  
ACCESSION NUMBER: 1973:545978 CAPLUS  
DOCUMENT NUMBER: 79:145978  
ORIGINAL REFERENCE NO.: 79:23661a, 23664a  
TITLE: O,O-Dialkylthiophosphoric acid pseudochalcogen acyls  
INVENTOR(S): Koehler, Helmut; Gerats, Irntraut; Eichler, Gerhard;  
Kochmann, Werner  
SOURCE: Ger. (East), 14 pp.  
CODEN: GEXXA8  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	DD 95374	A1	19730212	DD 1971-156303	19710705 <--
	PRIORITY APPLN. INFO.:			DD 1971-156303	A1 19710705
AB	(MeO)2P(S)N(CN)CH2CO2R (I) and/or (MeO)2P:(NCN)SCH2CO2R (II) (R = Me or Et), prepared by reacting (MeO)2P(S)NNAcN with XCH2CO2R (X = Br or Cl), gave 95.0, 52.5 and 69.0% mortality for R = Me and 92.5, 51.0 and 55.0% for R = Et at 0.01, 1.0 and 0.05 weight % concentration, resp., against Musca domestica, Sitophilus granarius and Tetranychus urticae, resp. Analogs of I and II wherein the CO2R group was replaced by CONH2 and CONHMe, and (MeO)2P(S)C(CN)CH2COR and (MeO)2P[: C(CN)2]SCH2COR (R = NHMe or OMe) were also prepared				
IT	50605-40-8P			RL: SPN (Synthetic preparation); PREP (Preparation)	
	50605-40-8			(preparation of)	
RN	50605-40-8			CAPLUS	
CN	Propanoic acid, 3,3-dicyano-3-(dimethoxyphosphinothioyl)-, methyl ester (CA INDEX NAME)				

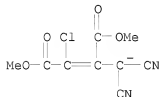


L5 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1964:484793 CAPLUS  
 DOCUMENT NUMBER: 61:84793  
 ORIGINAL REFERENCE NO.: 61:14826g-h,14827a-c  
 TITLE: 1-Halo-1,2,3,3-tetra(negatively substituted)propanes and their salts  
 INVENTOR(S): Martin, Elmore L.  
 PATENT ASSIGNEE(S): E. I. du Pont de Nemours & Co.  
 SOURCE: 6 pp.  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3133084		19640512	US	19600624 <--
PRIORITY APPLN. INFO.:			US	19600624

AB Comps. of the general formula [XC(Z):C(Y)C(A)R]- M+ (I), where A, R, Y, Z are electron withdrawing groups such as CN, CO<sub>2</sub>Et, Bz, or SO<sub>2</sub>Ph, X is Cl or F, and M is H, Na, K, or a substituted ammonium ion, are dyes for natural and synthetic fibers. Thus, H<sub>2</sub>C(CN)<sub>2</sub> 79 in tetrahydrofuran (II) 220 was added with stirring to a dispersion 52 of 51.2% NaH in mineral oil and II 660 at 5-10° during 15 min., the mixture stirred 30 min., then dichlorofumararonitrile 44 in II 220 added during 15 min., II vacuum-distilled at 35-40°, the residual yellow solid dissolved in H<sub>2</sub>O 250, the pH adjusted to 8 with CO<sub>2</sub>, then Et<sub>4</sub>NBr 100 in H<sub>2</sub>O 200 parts added slowly with stirring, the mixture cooled to 5°, and the yellow crystals of I (A = R = Y = X = CN, Z = Cl, M = Et<sub>4</sub>N) (III) filtered, washed with 1% Et<sub>4</sub>NBr, and then H<sub>2</sub>O. The cake was dissolved in H<sub>2</sub>O 3500 at 100°, decolorizing carbon 10 added, the solution clarified, cooled to 5°, the long yellow needles filtered, washed with H<sub>2</sub>O and air-dried, giving 70 parts III, m. 129-31°, λ<sub>maximum</sub> 387 mμ, ε = 18,200 (MeOH) yellow on cellulose acetate and nylon, brownish yellow on wool and silk. Similarly, other I were prepared as tabulated below: X, Z, Y, A, R, M, % yield, m.p., color, λ (mμ) maximum, ε; Cl, PhN(CO)-2, CN, CN, Me<sub>4</sub>N, 31 230-5° (decompose), orange, 468, 12,200; Cl, CO<sub>2</sub>Me, CO<sub>2</sub>Me, CN, CN, Et<sub>4</sub>N, 82, 88-90°, yellow, 335, 29,400; Cl, CN, CN, CO<sub>2</sub>Et, CO<sub>2</sub>Et, H, 100, b1, 115-20°, yellow (Na salt), -, -, Cl, Bz, Bz, CN, CN, Me<sub>4</sub>N, 39, 210-12° (decompose), yellow, 416, 27,000; F, CF<sub>3</sub>, CF<sub>3</sub>, CN, CN, Pr<sub>4</sub>N, 81, 84-6°, yellow, -, -, Cl, CN, CN, CN, CN, Me<sub>4</sub>N, -, 217-18° (decompose), yellow, 386, 17,600; Cl, CN, CN, CN, CN, Pr<sub>4</sub>N, -, 74-6° (decompose), yellow, 386, 18,100; Cl, CN, CN, CN, CN, Et<sub>3</sub>NH, -, 63-5° (decompose), yellow, 387, 17,200; Cl, CN, CN, CN, CO<sub>2</sub>Et, Et<sub>4</sub>N, 56, 70-2°, yellow, 400, 15,700; Cl, CN, CN, CN,

SO2C6H4Me-4, Me,N, 73, 124-6° (decompose), yellow, 387, 17,000; Cl,  
CN, CN, CN, Bz, Me4N, -, 159-61°, yellow, 414, 17,100; Cl, CN, Me,  
CN, Bz, Et4N, 30 118-19°, yellow, 420, 16,200; Cl, CN, CN, CN, CN,  
Pr4N, -, 109-10°, yellow, 412, 17,600; Cl, CF3, CF3, CN, CN, Et4N,  
64, 84-5°, yellow, -, -; F, -CF2CF2-, CN, CN, Na, -, -, orange, -,  
-; Cl, CN, CN, Bz, Bz, Me4N, -, 167-9°, yellow, 422, 8000;  
Cl, CN, CN, CN, CONHPh, K, -, -, red, -, -; Cl, CN, CN, SO2Ph, SO2Ph,  
Me4N, -, -, yellow, -, -; Cl, CN, CN, Bz, CO2Et, H, 20, 97-8°,  
colorless, -, -; Cl, CN, CN, Bz, CO2Et, Na, yellow;  
IT 98469-37-5P, Ammonium, tetraethyl, 1,2-dicarboxy-1-chloro-3,3-  
dicyanopropenide, dimethyl ester  
RL: PREP (Preparation)  
(preparation of)  
RN 98469-37-5 CAPLUS  
CN Tetraethylammonium 1,2-dicarboxy-1-chloro-3,3-dicyanopropenide, dimethyl  
ester (7CI) (CA INDEX NAME)  
  
CM 1  
  
CRN 98469-36-4  
CMF C9 H6 Cl N2 O4



CM	2
CRN	66-40-0
CMF	C8 H20 N

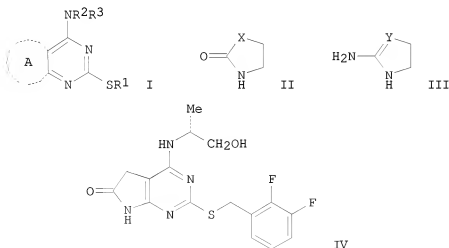


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L4 ANSWER 1 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
ACCESSION NUMBER: 2001:597989 CAPLUS  
DOCUMENT NUMBER: 135:166840  
TITLE: Preparation of pyrimidine compounds as modulators of  
chemokine receptor activity  
INVENTOR(S): Bonnett, Roger; Cagge, Peter; Hunt, Fraser; Walters,

PATENT ASSIGNEE(S): Lain; Willis, Paul  
 SOURCE: Astrazeneca Ab, Swed.  
 PCT Int. Appl., 52 pp.  
 CODEN: PIXXD2  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001058902	A1	20010816	WO 2001-SE245	20010207 <--
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW			
RW:	GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG			
GB 2359078	A	20010815	GB 2000-3019	20000211 <--
EP 1265899	A1	20021218	EP 2001-902950	20010207
R:	AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR			
JP 2003522189	T	20030722	JP 2001-558051	20010207
US 20030040523	A1	20030227	US 2002-203584	20020809
US 6958344	B2	20051025		
US 20050234077	A1	20051020	US 2005-36682	20050114
PRIORITY APPLN. INFO.:			GB 2000-3019	A 20000211
			WO 2001-SE245	W 20010207
			US 2002-203584	A1 20020809
OTHER SOURCE(S):	MARPAT 135:166840			
GI				

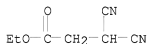


AB The title compds. [I; A = II, III (X = NH, CR18R19; Y = N, CR18; R18, R19 = H, alkyl, Ph); R1 = (un)substituted cycloalkyl, alkyl, alkenyl, etc.; R2, R3 = H, cycloalkyl, alkyl, etc.; NR2R3 = (un)substituted 3-8 membered ring optionally containing one or more atoms selected from O, S, NH, etc.], useful in treating an inflammatory disease such as psoriasis and COPD, were prepared E.g., a multi-step synthesis of the 6H-pyrrolo[2,3-d]pyrimidin-6-one IV was given. The compds. I were found to have IC50 of < 10 nM against CXCR2 receptor binding.

IT 224637-77-8  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (preparation of pyrimidine compds. as modulators of chemokine receptor activity)

RN 224637-77-8 CAPLUS

CN Propanoic acid, 3,3-dicyano-, ethyl ester (CA INDEX NAME)



REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 2 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2001:347933 CAPLUS

DOCUMENT NUMBER: 135:122094

TITLE: Cyclopropanation of benzylidenemalononitrile with dialkoxycarbenes and free radical rearrangement of the cyclopropanes

AUTHOR(S): Merkley, Nadine; Venneri, Paul C.; Warkentin, John

CORPORATE SOURCE: Department of Chemistry, McMaster University, Hamilton, ON, L8S 4M1, Can.

SOURCE: Canadian Journal of Chemistry (2001), 79(3), 312-318

CODEN: CJCHAG; ISSN: 0008-4042

PUBLISHER: National Research Council of Canada

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 135:122094

AB Thermolysis of 2-cinnamyloxy-2-methoxy-5,5-dimethyl-A3-1,3,4-oxadiazoline (1a) and the analogous 2-benzyloxy-2-methoxy compound (1b) at 110°C, in benzene containing benzylidenemalononitrile, afforded products of apparent regiospecific addition of methoxycarbonyl and cinnamyl (or benzyl) radicals to the double bond. When the thermolysis of 1a was run with added TEMPO, methoxycarbonyl and cinnamyl radicals were captured. Thermolysis of the 2,2-dibenzyloxy analog (1c) in the presence of benzylidenemalononitrile gave an adduct that is formally the product of addition of benzyloxycarbonyl and benzyl radicals to the double bond. In this case, a radical addition mechanism could be ruled out, because the rate constant for decarboxylation of benzyloxycarbonyl radicals is very large. A mechanism that fits all of the results is predominant cyclopropanation of benzylidenemalononitrile by the dialkoxycarbenes derived from the oxadiazolines, in competition with fragmentation of the carbenes to radical pairs. The cyclopropanes so formed then undergo homolytic



ring-opening to the appropriate diradicals. Subsequent  $\beta$ -scission of the diradicals to afford radical pairs, and coupling of those pairs, gives the final products. Thus, both carbene and radical chemical are involved in the overall processes.

IT 351207-62-0P 351207-63-1P 351207-65-3P

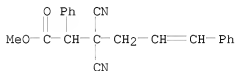
351207-66-4P 351207-67-5P

RL: SPN (Synthetic preparation); PREP (Preparation)

(cyclopropanation of benzylidenemalononitrile with dialkoxycarbenes and free radical rearrangement of the cyclopropanes)

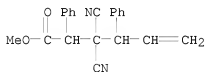
RN 351207-62-0 CAPLUS

CN Benzeneacetic acid,  $\alpha$ -(1,1-dicyano-4-phenyl-3-butenyl)-, methyl ester (9CI) (CA INDEX NAME)



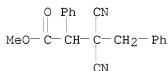
RN 351207-63-1 CAPLUS

CN Benzenebutanoic acid,  $\beta,\beta$ -dicyano- $\gamma$ -ethenyl- $\alpha$ -phenyl-, methyl ester (CA INDEX NAME)



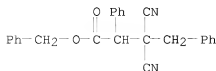
RN 351207-65-3 CAPLUS

CN Benzenebutanoic acid,  $\beta,\beta$ -dicyano- $\alpha$ -phenyl-, methyl ester (CA INDEX NAME)

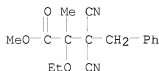


RN 351207-66-4 CAPLUS

CN Benzenebutanoic acid,  $\beta,\beta$ -dicyano- $\alpha$ -phenyl-, phenylmethyl ester (CA INDEX NAME)



RN 351207-67-5 CAPLUS  
 CN Benzenebutanoic acid,  $\beta,\beta$ -dicyano- $\alpha$ -ethoxy- $\alpha$ -methyl-, methyl ester (CA INDEX NAME)



REFERENCE COUNT: 20 THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 3 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2000:310730 CAPLUS

DOCUMENT NUMBER: 133:104844

TITLE: New aspects of Knoevenagel condensation and Michael addition reactions on alkaline carbonates

AUTHOR(S): Aramendia, Maria A.; Borau, Victoriano; Jimenez, Cesar; Marinas, Jose M.; Romero, Francisco J.

CORPORATE SOURCE: Department of Organic Chemistry, Faculty of Sciences, Cordoba University, Cordoba, E-14004, Spain

SOURCE: Chemistry Letters (2000), (5), 574-575

CODEN: CMLTAG; ISSN: 0366-7022

PUBLISHER: Chemical Society of Japan

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 133:104844

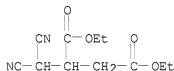
AB The Knoevenagel condensation of malononitrile with benzaldehyde on  $\text{K}_2\text{CO}_3$ ,  $\text{Rb}_2\text{CO}_3$  and  $\text{Cs}_2\text{CO}_3$  gave the condensation product benzylidenemalononitrile but the reaction proceeded to the hydrogenated product benzylmalononitrile. Also, the Michael addition of malononitrile to certain double bonds occurs in the presence of  $\text{K}_2\text{CO}_3$ .

IT 82584-86-9P

RL: SPN (Synthetic preparation); PREP (Preparation)  
 (potassium carbonate catalyzed Michael addition reactions of malononitrile with alkenes)

RN 82584-86-9 CAPLUS

CN Butanedioic acid, (dicyanomethyl)-, diethyl ester (9CI) (CA INDEX NAME)



REFERENCE COUNT: 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

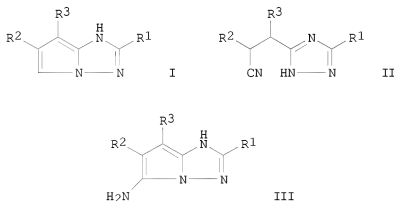
L4 ANSWER 4 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2000:199325 CAPLUS

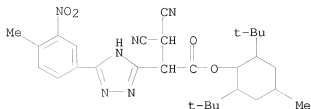
DOCUMENT NUMBER: 132:237096

TITLE: Preparation of 1H-pyrrolo-[1,2-b][1,2,4]triazole  
 INVENTOR(S): Morita, Kensuke  
 PATENT ASSIGNEE(S): Fuji Photo Film Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokyo Koho, 15 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2000086662	A	20000328	JP 1998-265064	19980918 <--
PRIORITY APPLN. INFO.:			JP 1998-265064	19980918
OTHER SOURCE(S):	CASREACT	132:237096; MARPAT	132:237096	
GI				



AB Title compds. I (R1-R3 = H, substituent) are prepared from triazoles II (R1-R3 = H, substituent) via III (R1-R3 = H, substituent).  
 3-(Tert-butylphenyl)-5-[[4-methyl-2,6-di-tert-butylhexyloxycarbonyl]bromomethyl]-1H-1,2,4-triazole was reacted with malononitrile in dimethylacetamide in the presence of NaOMe/MeOH under reflux for 30 min and reacted in the presence of CuCl in PhMe-hexane mixture under reflux for 3 h to give 91% III (R1 = 4-tert-butylphenyl; R2 = cyano, R3 = 4-methyl-2,6-di-tert-butylhexyloxycarbonyl), which was reacted with isoamyl nitrite in iso-Pr alc. at 50° for 10 h to give 40% I (R1-R3 = same as above).  
 IT 259266-71-2  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (preparation of pyrrolotriazoles from cyanoethyltriazoles)  
 RN 259266-71-2 CAPLUS  
 CN 1H-1,2,4-Triazole-3-acetic acid,  $\alpha$ -(dicyanomethyl)-5-(4-methyl-3-nitrophenyl)-, 2,6-bis(1,1-dimethylethyl)-4-methylcyclohexyl ester (CA INDEX NAME)

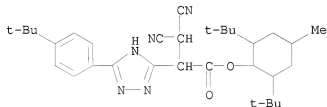


IT 259266-70-1P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation of pyrrolotriazoles from cyanoethyltriazoles)

RN 259266-70-1 CAPLUS

CN 1H-1,2,4-Triazole-3-acetic acid,  $\alpha$ -(dicyanomethyl)-5-[4-(1,1-dimethylethyl)phenyl]-, 2,6-bis(1,1-dimethylethyl)-4-methylcyclohexyl ester (CA INDEX NAME)

L4 ANSWER 5 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2000:198039 CAPLUS

DOCUMENT NUMBER: 132:238369

TITLE: 1H-Pyrrolo[1,2-b][1,2,4]triazole derivatives and their manufacture

INVENTOR(S): Morita, Kensuke

PATENT ASSIGNEE(S): Fuji Photo Film Co., Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 14 pp.

CODEN: JKXXAF

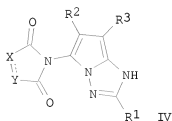
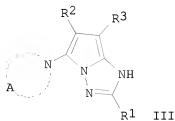
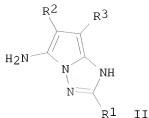
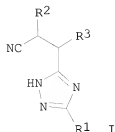
DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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JP 2000086661	A	20000328	JP 1998-265059	19980918 <--
PRIORITY APPLN. INFO.:			JP 1998-265059	19980918
OTHER SOURCE(S):	MARPAT	132:238369		
GI				



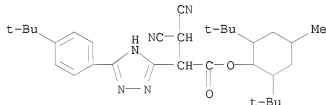
AB The derivs. III and IV, useful for photog. couplers, physiol. active substances, etc., are manufactured from triazole derivs. I via intermediates II (R1-3 = H, substituent; A = non-metal atomic group to form azole ring with N; X, Y = non-metal atom to form 5-membered ring with CONCO). Thus, I (R1 = p-tert-BuC6H4; R2 = CN; R3 = 2,6-di-tert-butyl-4-methylcyclohexyloxycarbonyl) was treated with CuCl to give 91% II, 9.42 mmol of which was treated with 11.3 mmol acetylacetone in benzene in the presence of AcOH to give 8.20 mmol III (azole ring = 3,4-dimethyl-1-pyrrolyl).

IT 259266-70-1P

RL: IMF (Industrial manufacture); RCT (Reactant); PREP (Preparation); RACT (Reactant or reagent)  
(manufacture of pyrrolo[1,2-a]triazole derivs.)

RN 259266-70-1 CAPLUS

CN 1H-1,2,4-Triazole-3-acetic acid,  $\alpha$ -(dicyanomethyl)-5-[4-(1,1-dimethylethyl)phenyl]-, 2,6-bis(1,1-dimethylethyl)-4-methylcyclohexyl ester (CA INDEX NAME)



L4 ANSWER 6 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
ACCESSION NUMBER: 2000:141061 CAPLUS  
DOCUMENT NUMBER: 132:278722

TITLE: Spontaneous addition of active methine compounds to enol ethers and  $\alpha,\beta$ -unsaturated ketones in aprotic polar solvent

AUTHOR(S): Yokozawa, Tsutomu; Oishi, Motoi; Tanaka, Yasukazu

CORPORATE SOURCE: Department of Applied Chemistry, Kanagawa University, Kanagawa-ku Yokohama, 221-8686, Japan

SOURCE: Journal of Organic Chemistry (2000), 65(6), 1895-1897  
CODEN: JOCEAH; ISSN: 0022-3263

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

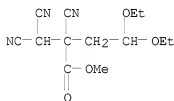
OTHER SOURCE(S): CASREACT 132:278722

AB Addition of (EtO)2CHCH2CXYCH(CN)2 (I, X = Y = cyano; X = cyano, Y = CO2Me; X = Y = CO2Me) to enol ethers and  $\alpha,\beta$ -unsatd. ketones in DMF at room temp is reported. Thus, reacting I (X = Y = cyano) with H2C:CHOEt gave (EtO)2CHCH2C(CN)2CH(OEt)Me in 63% yield. This reaction illustrates that the electron-withdrawing groups at the  $\beta$ -positions of the active methine group having the ones at the  $\alpha$  and  $\beta$  positions were strongly affected on the acidity of I.

IT 184092-93-1 189348-52-5  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(addition of methine compds. to enol ethers and  $\alpha,\beta$ -unsatd. ketones)

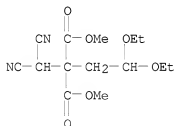
RN 184092-93-1 CAPLUS

CN Butanoic acid, 2-cyano-2-(dicyanomethyl)-4,4-diethoxy-, methyl ester (CA INDEX NAME)



RN 189348-52-5 CAPLUS

CN Propanedioic acid, (dicyanomethyl)(2,2-diethoxyethyl)-, dimethyl ester (9CI) (CA INDEX NAME)



IT 264142-31-6P 264142-33-8P 264142-35-0P  
264142-37-2P 264142-39-4P 264142-40-7P

10/923,271

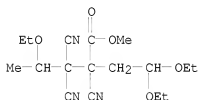
264142-41-8P 264142-43-0P 264142-45-2P

RL: SPN (Synthetic preparation); PREP (Preparation)

(addition of methine compds. to enol ethers and  $\alpha,\beta$ -unsatd. ketones)

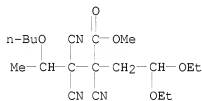
RN 264142-31-6 CAPLUS

CN Pentanoic acid, 2,3,3-tricyano-2-(2,2-diethoxyethyl)-4-ethoxy-, methyl ester (CA INDEX NAME)



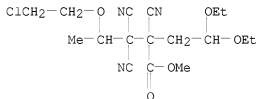
RN 264142-33-8 CAPLUS

CN Pentanoic acid, 4-butoxy-2,3,3-tricyano-2-(2,2-diethoxyethyl)-, methyl ester (CA INDEX NAME)



RN 264142-35-0 CAPLUS

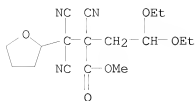
CN Pentanoic acid, 4-(2-chloroethoxy)-2,3,3-tricyano-2-(2,2-diethoxyethyl)-, methyl ester (CA INDEX NAME)



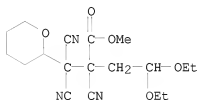
RN 264142-37-2 CAPLUS

CN 2-Furanpropanoic acid,  $\alpha,\beta,\beta$ -tricyano- $\alpha$ -(2,2-diethoxyethyl)tetrahydro-, methyl ester (CA INDEX NAME)

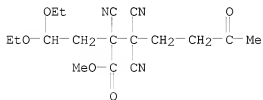
10/923,271



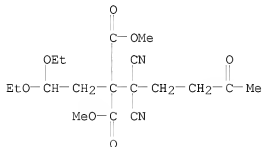
RN 264142-39-4 CAPLUS  
CN 2H-Pyran-2-propanoic acid,  $\alpha,\beta,\beta$ -tricyano- $\alpha$ -(2,2-diethoxyethyl)tetrahydro-, methyl ester (CA INDEX NAME)



RN 264142-40-7 CAPLUS  
CN Heptanoic acid, 2,3,3-tricyano-2-(2,2-diethoxyethyl)-6-oxo-, methyl ester (CA INDEX NAME)



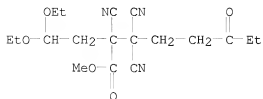
RN 264142-41-8 CAPLUS  
CN Propanedioic acid, (1,1-dicyano-4-oxopentyl)(2,2-diethoxyethyl)-, dimethyl ester (9CI) (CA INDEX NAME)



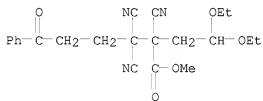
RN 264142-43-0 CAPLUS  
CN Octanoic acid, 2,3,3-tricyano-2-(2,2-diethoxyethyl)-6-oxo-, methyl ester



## (CA INDEX NAME)



RN 264142-45-2 CAPLUS

CN Benzenehexanoic acid,  $\alpha,\beta,\beta$ -tricyano- $\alpha$ -(2,2-diethoxyethyl)- $\epsilon$ -oxo-, methyl ester (CA INDEX NAME)

REFERENCE COUNT: 25 THERE ARE 25 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 7 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2000:139178 CAPLUS

DOCUMENT NUMBER: 132:180579

TITLE: Preparation of 1H-pyrrolo[1,2-b][1,2,4]triazol-5-ylamines

INVENTOR(S): Morita, Kensuke

PATENT ASSIGNEE(S): Fuji Photo Film Co., Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 15 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

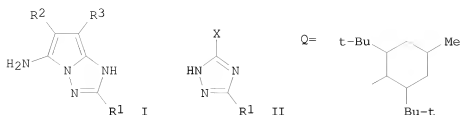
LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2000063382	A	20000229	JP 1998-232925	19980819 <--
PRIORITY APPLN. INFO.:			JP 1998-232925	19980819
OTHER SOURCE(S):		CASREACT 132:180579; MARPAT 132:180579		

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AB Title compds. I (R1-R3 = H, substituent), useful as intermediates for physiol. active substances, photog. couplers, dyes, etc., are prepared from triazoles II (X = CHR3CHR2CN; R1-R3 = same as I). II (R1 = C6H4Bu-t-p, X = CHBrCO2Q) was treated with malononitrile and MeONa in DMF-MeOH under ice-cooling for 30 min and heated in the presence of CuCl in PhMe-hexane under reflux for 3 h to give 91% I (R1 = C6H4Bu-t-p, R2 = cyano, R3 = CO2Q).

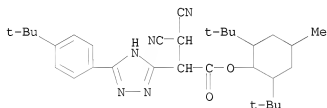
IT 259266-70-1P 259266-71-2P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation of pyrrolotriazolylamines as intermediates for physiol. active substances, dyes, and photog. couplers)

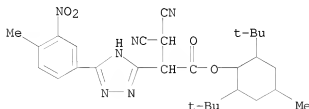
RN 259266-70-1 CAPLUS

CN 1H-1,2,4-Triazole-3-acetic acid,  $\alpha$ -(dicyanomethyl)-5-[4-(1,1-dimethylethyl)phenyl]-, 2,6-bis(1,1-dimethylethyl)-4-methylcyclohexyl ester (CA INDEX NAME)

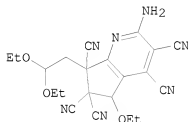


RN 259266-71-2 CAPLUS

CN 1H-1,2,4-Triazole-3-acetic acid,  $\alpha$ -(dicyanomethyl)-5-[4-methyl-3-nitrophenyl]-, 2,6-bis(1,1-dimethylethyl)-4-methylcyclohexyl ester (CA INDEX NAME)

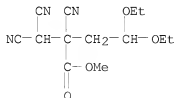


L4 ANSWER 8 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1999:380253 CAPLUS  
 DOCUMENT NUMBER: 131:170252  
 TITLE: Tandem dimerization and double annulation of  
 3,3,4,4-tetracyanobutanal acetal. Synthesis of a  
 bicyclic 2-aminopyridine derivative  
 AUTHOR(S): Yokozawa, Tsutomu; Nishikata, Akira; Kimura, Takamasa;  
 Shimizu, Kazuki; Takehana, Tomoyuki  
 CORPORATE SOURCE: Department of Applied Chemistry, Kanagawa University,  
 Yokohama, 221-8686, Japan  
 SOURCE: Tetrahedron Letters (1999), 40(25),  
 4707-4710  
 CODEN: TELEAY; ISSN: 0040-4039  
 PUBLISHER: Elsevier Science Ltd.  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 131:170252  
 GI



II

AB 3,3,4,4-Tetracyanobutanal acetal (I), which is easily obtained from  
 tetracyanoethylene, Et vinyl ether, and ethanol, yielded 2-aminopyridine  
 derivative II fused with cyclopentane in one pot in the presence of pyridine.  
 On the basis of several expts., the proposed mechanism involves the  
 Michael reaction of I with the diene generated by the elimination of  
 hydrogen cyanide and ethanol from I, followed by double intramol.  
 nucleophilic addns. to the cyano groups.  
 IT 184092-93-1  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (preparation of bicyclic aminopyridine by tandem dimerization-cyclization of  
 tetracyanobutanal acetal)  
 RN 184092-93-1 CAPLUS  
 CN Butanoic acid, 2-cyano-2-(dicyanomethyl)-4,4-diethoxy-, methyl ester (CA  
 INDEX NAME)



REFERENCE COUNT: 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 9 OF 68 CAPLUS COPYRIGHT 2008 ACS ON STN

ACCESSION NUMBER: 1999:142792 CAPLUS

DOCUMENT NUMBER: 130:360507

TITLE: An N2S2 Bifunctional Chelator for Technetium-99m and Rhenium: Complexation, Conjugation, and Epimerization to a Single Isomer

AUTHOR(S): Luyt, Leonard G.; Jenkins, Hilary A.; Hunter, Duncan H.

CORPORATE SOURCE: Department of Chemistry, University of Western Ontario, London, ON, N6A 5B7, Can.

SOURCE: Bioconjugate Chemistry (1999), 10(3), 470-479

CODEN: BCCHEJ; ISSN: 1043-1802

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

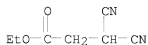
LANGUAGE: English

AB A bifunctional chelator HO2CCH(CH2NHCOCH2SH)2 6 (H3L) was prepared bearing an N2S2 core for binding Re or Tc and a carboxylic acid group for conjugation to amino groups of biomols. Complexation of 6 with Re(V) resulted in two kinetic isomers, anti- and syn-[ReO(HL)]- 7, being formed in approx. equal amts. Epimerization with 0.5M NaOH yields a single isomer anti-7, as determined by NMR spectroscopy and single-crystal x-ray anal. [99mTcO(HL)]- was prepared at the tracer level by reaction of the ligand with 99mTcO4-, SnCl2 and Na gluconate giving a mixture of two isomers, but showing a preference for the anti isomer. Chelation in the presence of 1 M NaOH results in anti-8 being formed as the sole product. The bifunctional ability of the ligand was explored by amide formation with (S)- $\alpha$ -phenethylamine, either by direct DCC coupling or through the RO2CCH(CH2NHCOCH2S(Tr))2 9 (R = succinimidyl) intermediate. The deprotected bioconjugate PhCHMeNHOCCH(CH2NHCOCH2SH)2 11 (H2L1) was complexed with Re, yielding similar amts. of two isomeric Re complexes, anti- and syn-12, which were isolated and characterized by NMR spectroscopy. Treatment of the kinetic mixture of anti- and syn-[ReOL1]- 12 with 1 M NaOH resulted in quant. conversion to a single Re complex anti-12. With 99mTc in 0.1M NaOAc, bioconjugate 11 yielded anti- and sym-[99mTcOL1]- 13 in a 2:1 ratio, resp. In contrast, complexation in the presence of 1 M NaOH gave only one 99mTc complex, assigned the structure anti-13.

IT 224637-77-8P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(preparation and reactant for preparation of rhenium and technetium bis(thioacetamidomethyl)propionate and bis(thioacetamidomethyl)propanamide complexes)

RN 224637-77-8 CAPLUS

CN Propanoic acid, 3,3-dicyano-, ethyl ester (CA INDEX NAME)

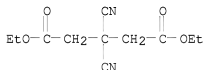


REFERENCE COUNT: 24 THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS

## RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 10 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1999:142376 CAPLUS  
 DOCUMENT NUMBER: 130:239567  
 TITLE: Diazaspirononanium salt for use as template for zeolite synthesis  
 INVENTOR(S): Kubota, Yoshihiro; Sugi, Yoshihiro  
 PATENT ASSIGNEE(S): Showa Denko K. K., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 9 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

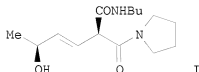
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 11060577	A	19990302	JP 1997-220414	19970815 <--
PRIORITY APPLN. INFO.:			JP 1997-220414	19970815
OTHER SOURCE(S):	MARPAT	130:239567		
AB	Claimed template is a salt of substituted 2,7-diazaspiro[4,4]nonanium. Hydrothermal synthesis of a zeolite by bringing a silica source and/or an alumina source into contact with the zeolite is also claimed. ZSM-12 zeolites having crystal size of a major axis $\geq 50 \mu\text{m}$ are also claimed.			
IT	77415-69-1P RL: IMF (Industrial manufacture); RCT (Reactant); PREP (Preparation); RACT (Reactant or reagent) (reaction of; diazaspirononanium salts as templates for manufacture of ZSM-12 zeolites having large crystal size)			
RN	77415-69-1 CAPLUS			
CN	Pentanedioic acid, 3,3-dicyano-, diethyl ester (9CI) (CA INDEX NAME)			



L4 ANSWER 11 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1999:118566 CAPLUS  
 DOCUMENT NUMBER: 130:237421  
 TITLE: Stereoselective synthesis of alkenylated malonic diamide using masked acyl cyanide  
 AUTHOR(S): Nemoto, Hisao; Ibaragi, Toru; Bando, Masahiko; Kido, Masaru; Shibuya, Masayuki  
 CORPORATE SOURCE: Faculty of Pharmaceutical Sciences, the University of Tokushima, Tokushima, 770-8505, Japan  
 SOURCE: Tetrahedron Letters (1999), 40(7), 1319-1322  
 CODEN: TELEAY; ISSN: 0040-4039  
 PUBLISHER: Elsevier Science Ltd.  
 DOCUMENT TYPE: Journal

10/923,271

LANGUAGE: English  
OTHER SOURCE(S): CASREACT 130:237421  
GI



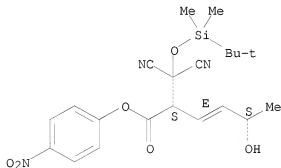
AB A highly stereoselective synthesis of an alkenylated malonic diamide (I) starting from a  $\gamma,\delta$ -epoxy  $\alpha,\beta$ -unsatd. carboxamide was accomplished using masked acyl cyanide (protected hydroxymalonitrile) via palladium-catalyzed regio- and stereoselective carbon-carbon bond formation.

IT 221219-72-3P 221219-73-4P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(stereoselective synthesis of alkenylated malonic diamide using masked acyl cyanide)

RN 221219-72-3 CAPLUS

CN 3-Hexenoic acid, 2-[dicyano[[ (1,1-dimethylethyl)dimethylsilyl]oxy]methyl]-5-hydroxy-, 4-nitrophenyl ester, (2R,3E,5R)-rel- (CA INDEX NAME)

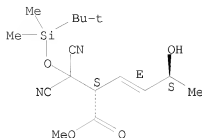
Relative stereochemistry.  
Double bond geometry as shown.



RN 221219-73-4 CAPLUS

CN 3-Hexenoic acid, 2-[dicyano[[ (1,1-dimethylethyl)dimethylsilyl]oxy]methyl]-5-hydroxy-, methyl ester, (2R,3E,5R)-rel- (CA INDEX NAME)

Relative stereochemistry.  
Double bond geometry as shown.



REFERENCE COUNT: 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 12 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1998:402031 CAPLUS

DOCUMENT NUMBER: 129:122635

TITLE: The reaction of phenacylmalononitrile with hydrazines: synthesis of new pyridazinones and pyrazolo[1,5-a]pyrimidines

AUTHOR(S): Elnagdi, Mohamed Hilmy; El-Ghamry, Ibrahim; Kandeel, Ezz; Abdel Rahman, A. H.; Al-Naggar, Abdul Aziz; Amer, Samir; Riad, Mohamed

CORPORATE SOURCE: Department of Chemistry, Faculty of Science, University of Kuwait, Safat, 13060, Kuwait

SOURCE: Gazzetta Chimica Italiana (1997), 127(12), 791-794

CODEN: GCITA9; ISSN: 0016-5603

PUBLISHER: Societa Chimica Italiana

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 129:122635

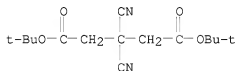
AB The reaction of phenacylmalononitrile with hydrazine hydrate affords a mixture of 3,5-diamino-4-phenacylpyrazole (I), and 6-oxo-3-phenyl-1,4,5,6-tetrahydropyridazine-5-carbonitrile. The reaction of I with a variety of reagents, that enabled the synthesis of some new pyrazolo[1,5-a]pyrimidine derivs., is described.

IT 210347-41-4P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(synthesis of pyridazinones and pyrazolo[1,5-a]pyrimidines)

RN 210347-41-4 CAPLUS

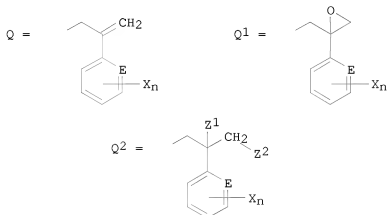
CN Pentanedioic acid, 3,3-dicyano-, bis(1,1-dimethylethyl) ester (9CI) (CA INDEX NAME)



REFERENCE COUNT: 15 THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 13 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1998:76233 CAPLUS  
 DOCUMENT NUMBER: 128:177233  
 TITLE: Malononitrile derivatives and herbicides containing them  
 INVENTOR(S): Hosokawa, Akemi; Ikeda, Osamu  
 PATENT ASSIGNEE(S): Mitsubishi Chemical Industries Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 18 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 10029966	A	19980203	JP 1996-187796	19960717 <---
PRIORITY APPLN. INFO.: OTHER SOURCE(S): GI	MARPAT 128:177233			19960717



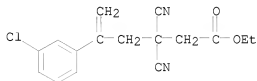
- AB The derivs. are represented by R1R2C(CN)2 [I; R1 = H, C1-6 alkyl, C4-7 cycloalkyl, C2-6 alkenyl, C2-6 alkynyl, C1-6 haloalkyl, C2-6 haloalkenyl, C2-6 haloalkynyl, C2-8 alkoxyalkyl, C3-6 alkoxyalkonylalkyl, C2-4 cyanoalkyl, C1-6 hydroxyalkyl, C2-7 alkylamido, C7-9 aralkyl, C8-12 arylcarbonylalkyl, (un)substituted Ph, (un)substituted pyridyl, (un)substituted thiazolyl, CR3R4A; A = (un)substituted Ph, pyridyl, thiazolyl; R3-4 = H, Me; R2 = Q, Q1, Q2; E = CH, N; X = halo, C1-4 alkyl, C1-3 haloalkyl, NO2, C1-8 haloalkoxy, (un)substituted benzyloxy, pyridyloxy; n = 0-2; Z1-2 = OH, halo, C1-4 alkylsulfonyloxy, (un)substituted phenylsulfonyloxy]. The herbicides contain I as active ingredients. I (R1 = H, R2 = Q, E = CH, X = 3-Me, 5-Me) showed 91-100% herbicidal activity against Echinochloa oryzicola, Monochoria vaginalis, and Scirpus juncoides.
- IT 203127-60-0P 203127-94-0P  
 RL: AGR (Agricultural use); BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); SPN (Synthetic



preparation); BIOL (Biological study); PREP (Preparation); USES (Uses)  
(preparation of malononitrile derivs. as herbicides)

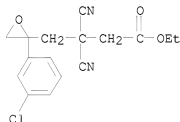
RN 203127-60-0 CAPLUS

CN Benzenepentanoic acid, 3-chloro- $\beta,\beta$ -dicyano- $\delta$ -methylene-,  
ethyl ester (CA INDEX NAME)



RN 203127-94-0 CAPLUS

CN Oxiranebutanoic acid, 2-(3-chlorophenyl)- $\beta,\beta$ -dicyano-, ethyl  
ester (9CI) (CA INDEX NAME)



L4 ANSWER 14 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1997:731010 CAPLUS

DOCUMENT NUMBER: 127:346753

TITLE: Synthesis of terpolymers by spontaneous  
copolymerization of the cyclobutane adducts of  
electron-acceptor olefins and vinyl ether with  
2-oxazolines

AUTHOR(S): Yokozawa, Tsutomu; Tagami, Masato; Takehana, Tomoyuki;  
Suzuki, Tadashi

CORPORATE SOURCE: Dep. Appl. Chem., Kanagawa Univ., Yokohama, 221, Japan

SOURCE: Tetrahedron (1997), 53(45), 15603-15616

CODEN: TETRAB; ISSN: 0040-4020

PUBLISHER: Elsevier

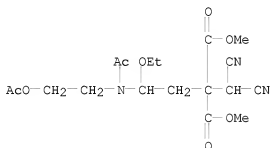
DOCUMENT TYPE: Journal

LANGUAGE: English

AB Spontaneous copolymns. of the cyclobutane adducts of strong donors olefins  
and strong acceptor olefins, 1,1,2,2-tetracyano-3-ethoxycyclobutene (I)  
and di-Me 2,2-dicyano-3-ethoxycyclobutane-1,1-dicarboxylate (II), with  
2-oxazolines are described. In the reaction of II with 2-methyloxazoline  
(III), the alternating copolymer of II and III, the 1:1:1 periodic  
terpolymer of di-Me 1,1-dicyanoethylene-2,2-dicarboxylate, vinyl ether,  
and III, was obtained. Cyclobutane I also reacted with III to yield  
copolymer rich in I.

IT 198274-09-8P

RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation and characterization of)  
 RN 198274-09-8 CAPLUS  
 CN Propanedioic acid, [2-[acetyl[2-(acetyloxy)ethyl]amino]-2-ethoxyethyl](dicyanomethyl)-, dimethyl ester (9CI) (CA INDEX NAME)



REFERENCE COUNT: 37 THERE ARE 37 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 15 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1997:513581 CAPLUS

DOCUMENT NUMBER: 127:184884

TITLE: Multinuclear cluster complexes as diagnostic imaging contrast agents

INVENTOR(S): Droege, Michael; Yu, Shi-Bao; Sanderson, William; Bacon, Edward; Delecki, Daniel; Earley, William; Ye, Naidong

PATENT ASSIGNEE(S): Nycomed Salutar, Inc., USA

SOURCE: PCT Int. Appl., 51 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9726921	A2	19970731	WO 1997-GB211	19970123 <--
WO 9726921	A3	19971023		
W:	AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN			
RW:	KE, LS, MW, SD, SZ, UG, AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG			
CA 2241190	A1	19970731	CA 1997-2241190	19970123 <--
AU 9714523	A	19970820	AU 1997-14523	19970123 <--
EP 876161	A2	19981111	EP 1997-901180	19970123 <--
EP 876161	B1	20011121		
R:	DE, DK, ES, FR, GB, IT, SE, IE, FI			
CN 1208353	A	19990217	CN 1997-191757	19970123 <--
HU 9901488	A2	19990830	HU 1999-1488	19970123 <--

BR 9707300	A	19991228	BR 1997-7300	19970123 <--
JP 2000515850	T	20001128	JP 1997-526668	19970123 <--
NO 9803371	A	19980722	NO 1998-3371	19980722 <--
PRIORITY APPLN. INFO.:			GB 1996-1340	A 19960123
			WO 1997-GB211	W 19970123

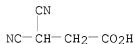
OTHER SOURCE(S): MARPAT 127:184884

AB Diagnostic imaging contrast media are claimed comprising a physiologically tolerable image contrast-enhancing complex, said complex comprising a pair of interconjugated multinuclear clusters, together with at least one pharmaceutical carrier or excipient. Included, for example, are multinuclear cluster complexes (M3)2L3 containing three metal atoms and L is a ligand. Clusters (M3)2L3 include M3 = M3SaOb where a = 1-4, b = 0-3 and a + b = 4, e.g., M3 = W3SO3. Ligands L include various polyaminocarboxylates and derivs. represented by general formula (R2)2N[(CHR4)mNR1]n(CHR4)mN(R2)2, e.g., N'-serinol-, N'-methyl-, and N'-benzyl-N,N,N',N'-diethylenetriaminetetraacetic acids, various N'-(polyhydroxyalkyl)-N'-methyldiethylenetriaminetetraacetic acids, 2-carboxymethylpropylenediaminetetraacetic acid, etc., for which preps. are given of these and other example ligands. Preparation of cluster compds., e.g., Na4[(W3SO3)2(EGTA)3] (EGTA = ethyleneglycol bis(2-aminoethyl ether)-N,N,N',N'-tetraacetate), from [W3SO3(H2O)9]Cl4 and the appropriate polyaminocarboxylic acid ligand, are described. The claimed preparation of [W3SO3(H2O)9]Cl4 comprises reaction of W(CO)6 and Na2S, followed by acidification of the product with at least 6 N HCl, and purification. A charged contrast medium complex may be post-complexed with, e.g., choline hydrochloride or N-methyl-N,N-bis(hydroxyethyl)ethylenediamine, to give a preferred neutral derivative. Pharmaceutically acceptable forms of the diagnostic imaging contrast media comprising said cluster complexes and dosages for the x-ray contrast media are briefly discussed.

IT 194083-97-1P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (for preparation of multinuclear tungsten aminocarboxylate cluster complexes as diagnostic imaging contrast agents)

RN 194083-97-1 CAPLUS

CN Propanoic acid, 3,3-dicyano- (CA INDEX NAME)



L4 ANSWER 16 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1997:414890 CAPLUS

DOCUMENT NUMBER: 127:144690

TITLE: Metabolism and disposition of the antifolate LY231514 in mice and dogs

AUTHOR(S): Woodland, J. M.; Barnett, C. J.; Dorman, D. E.; Gruber, J. M.; Shih, C.; Spangle, L. A.; Wilson, T. M.; Ehlhardt, W. J.

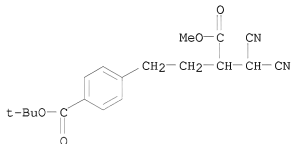
CORPORATE SOURCE: Lilly Res. Laboratories, USA

SOURCE: Drug Metabolism and Disposition (1997), 25(6), 693-700

CODEN: DMDSAI; ISSN: 0090-9556

PUBLISHER: Williams & Wilkins  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English

- AB The metabolism and disposition of LY231514 was studied in mice and dogs. LY231514 is a novel pyrrolopyrimidine-based multi-target antifolate (MTA) showing broad in vivo antitumor activity in mouse models and is currently in phase II human clin. trials. Doses (i.v.) of the compound showed high plasma levels, resulting in AUC values of 30-33 µg-hr/mL for mice and dogs after 20 and 7.5 mg/kg doses, resp. The compound was eliminated rapidly. Half-life values for mice and dogs were about 7 and 2 h, resp. In vitro plasma binding measured 56% in mice, 46% in dogs, and 81% in humans. Fecal elimination was the major excretion pathway in mice after single i.v. doses of [14C]LY231514. Urine constituted the major route of excretion in dogs. Parent LY231514 accounted for the majority of urinary radiocarbon in mice (90%) and dogs (68%). Minor metabolites were found in urine, but the amts. were too small to isolate or identify. Based on an earlier observation that LY231514 photodegraded to produce reaction products having similar retention times as these minor urinary isolates, a photo oxidation system was developed which in fact produced these metabolites. Subsequently, these photolytically produced materials were used as stds. to identify two novel in vivo metabolites formed by oxidation of the pyrrolo-pyrimidine ring system of LY231514. The oxidative transformations are similar to those observed for tryptophan and other indoles in that the pyrrole ring is oxidized to give an amide; further oxidation cleaves this ring, one ring carbon is lost, and a ketone is formed.
- IT 193265-49-5P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (antifolate drug LY231514 metabolism and pharmacokinetics in mice and dogs)
- RN 193265-49-5 CAPLUS
- CN Benzenebutanoic acid, α-(dicyanomethyl)-4-[(1,1-dimethylethoxy)carbonyl]-, methyl ester (CA INDEX NAME)



L4 ANSWER 17 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1997:330734 CAPLUS  
 DOCUMENT NUMBER: 127:34293  
 TITLE: The reactions of Wittig-Horner reagents with 1,3-dioxo- $\alpha$ -indanmalononitrile  
 AUTHOR(S): Boulos, Leila Sadek; Yakout, El-Sayed M. A.  
 CORPORATE SOURCE: National Research Centre, Cairo, Egypt  
 SOURCE: Heteroatom Chemistry (1997), 8(3), 253-257  
 CODEN: HETCE8; ISSN: 1042-7163

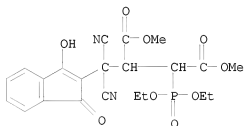
PUBLISHER: Wiley  
DOCUMENT TYPE: Journal  
LANGUAGE: English

AB Wittig-Horner reagents react with 1,3-dioxo- $\Delta^2,\alpha$ -indanmalononitrile to give phosphonate adducts. Structural reasoning for the new products was based on compatible anal. and spectral data (IR,  $^1\text{H}$ ,  $^{31}\text{P}$  NMR, and MS). The mechanism that accounts for the formation of the new adducts is discussed.

IT 190722-21-5P 190722-23-7P 190722-25-9P  
RL: SPN (Synthetic preparation); PREP (Preparation of)  
(preparation of)

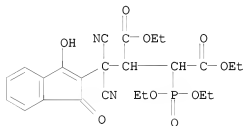
RN 190722-21-5 CAPLUS

CN Butanedioic acid, 2-[dicyano(3-hydroxy-1-oxo-1H-inden-2-yl)methyl]-3-(diethoxyphosphinyl)-, dimethyl ester (9CI) (CA INDEX NAME)



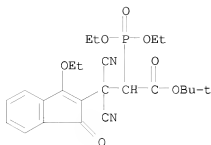
RN 190722-23-7 CAPLUS

CN Butanedioic acid, 2-[dicyano(3-hydroxy-1-oxo-1H-inden-2-yl)methyl]-3-(diethoxyphosphinyl)-, diethyl ester (9CI) (CA INDEX NAME)



RN 190722-25-9 CAPLUS

CN 1H-Indene-2-propanoic acid,  $\beta,\beta$ -dicyano- $\alpha$ -(diethoxyphosphinyl)-3-ethoxy-1-oxo-, 1,1-dimethylethyl ester (CA INDEX NAME)



REFERENCE COUNT: 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 18 OF 68 CAPLUS COPYRIGHT 2003 ACS on STN

ACCESSION NUMBER: 1997:274825 CAPLUS

DOCUMENT NUMBER: 126:317775

TITLE: Ring-opening polymerization of cyclobutane adduct of dimethyl 1,1-dicyanoethylene-2,2-dicarboxylate and ethyl vinyl ether

AUTHOR(S): Yokozawa, Tsutomu; Wakabayashi, Yuki; Kimura, Takamasa

CORPORATE SOURCE: Dep. Applied Chem., Kanagawa Univ., Yokohama, 221, Japan

SOURCE: Journal of Polymer Science, Part A: Polymer Chemistry (1997), 35(8), 1563-1570

CODEN: JPACEC; ISSN: 0887-624X

PUBLISHER: Wiley

DOCUMENT TYPE: Journal

LANGUAGE: English

AB For an extension of the work on the ring-opening polymns. of cyclobutane adducts of strong donor olefins and strong acceptor olefins yielding novel alternating copolymers of those olefins, the ring-opening polymerization of the cyclobutane adduct (I; di-Me 2,2-dicyano-3-ethoxy-1,1-cyclobutanedicarboxylate) of di-Me 1,1-dicyanoethylene-2,2-dicarboxylate (DDED) and Et vinyl ether (EVE) is investigated. I reacted with methanol and acetic acid at ambient temperature to yield the corresponding ring-opened adducts. Polymns. of I were carried out with anionic initiators, tertiary amines, ammonium halides, and Lewis acids, resp., according to the polymerization

methods of the cyclobutane adduct of tetracyanoethylene and EVE. All these polymerization catalysts except for ammonium halides were effective for

the polymerization of I, yielding alternating copolymers of DDED and EVE. The chain

transfer reactions of the polymerization with anionic initiators are also discussed on the basis of a model reaction.

IT 189348-52-5

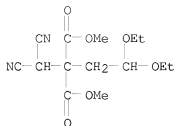
RL: RCT (Reactant); RACT (Reactant or reagent)

(model reaction for determination of mechanism; ring-opening polymerization of di-Me

2,2-dicyano-3-ethoxy-1,1-cyclobutanedicarboxylate)

RN 189348-52-5 CAPLUS

CN Propanedioic acid, (dicyanomethyl) (2,2-diethoxyethyl)-, dimethyl ester (9CI) (CA INDEX NAME)



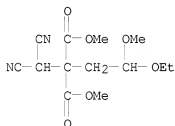
IT 189348-50-3P 189348-51-4P

RL: SPN (Synthetic preparation); PREP (Preparation)  
 (model reactions for polymerization; reactions of di-Me  
 2,2-dicyano-3-ethoxy-

1,1-cyclobutanedicarboxylate with acetic acid and methanol)

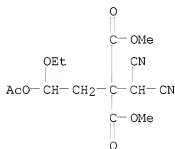
RN 189348-50-3 CAPLUS

CN Propanedioic acid, (dicyanomethyl) (2-ethoxy-2-methoxyethyl)-, dimethyl  
 ester (9CI) (CA INDEX NAME)



RN 189348-51-4 CAPLUS

CN Propanedioic acid, [2-(acetyloxy)-2-ethoxyethyl] (dicyanomethyl)-, dimethyl  
 ester (9CI) (CA INDEX NAME)



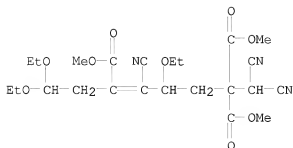
IT 189348-53-6P

RL: SPN (Synthetic preparation); PREP (Preparation)  
 (ring-opening polymerization of di-Me 2,2-dicyano-3-ethoxy-1,1-  
 cyclobutanedicarboxylate)

RN 189348-53-6 CAPLUS

CN 5-Octene-2,2,6-tricarboxylic acid, 1,1,5-tricyano-4,8,8-triethoxy-,

trimethyl ester (9CI) (CA INDEX NAME)



REFERENCE COUNT: 27 THERE ARE 27 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 19 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1996:672685 CAPLUS

DOCUMENT NUMBER: 126:8777

TITLE: Ring-Opening Polymerization of the Cyclobutane Adduct of Methyl Tricyanoethylenecarboxylate and Ethyl Vinyl Ether

AUTHOR(S): Yokozawa, Tsutomu; Tsuruta, Ei-ichi

CORPORATE SOURCE: Department of Applied Chemistry, Kanagawa University, Yokohama, 221, Japan

SOURCE: Macromolecules (1996), 29(25), 8053-8056

CODEN: MAMOBX; ISSN: 0024-9297

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The ring-opening polymns. of a cyclobutane adduct (I) of Me tricyanoethylenecarboxylate (MICE) and Et vinyl ether (EVE) are investigated. The adduct I reacted with acetic acid and ethanol at ambient temperature to yield the ring-opened corresponding adducts in good yields. I was polymerized with Lewis acids, anionic initiators, tertiary amines, and ammonium halides. All the catalysts except for ammonium halides were effective for the alternating polymerization similar to the polymerization

of cyclobutane adduct of TCNE and EVE.

IT 184092-92-0P 184092-93-1P

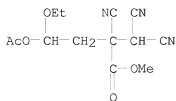
RL: SPN (Synthetic preparation); PREP (Preparation)

(ring-opening reactivity of cyclobutane adduct of Me tricyanoethylenecarboxylate with ethanol or acetic acid and Et vinyl ether)

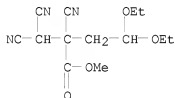
RN 184092-92-0 CAPLUS

CN Butanoic acid, 4-(acetyloxy)-2-cyano-2-(dicyanomethyl)-4-ethoxy-, methyl ester (CA INDEX NAME)





RN 184092-93-1 CAPLUS  
 CN Butanoic acid, 2-cyano-2-(dicyanomethyl)-4,4-diethoxy-, methyl ester (CA  
 INDEX NAME)



REFERENCE COUNT: 9 THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS  
 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 20 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1996:664756 CAPLUS  
 DOCUMENT NUMBER: 125:329472  
 TITLE: Preparation of ring-fused pyrimidine-containing amino  
 acid derivatives as antiprotozoan agents  
 INVENTOR(S): Horii, Toshihiro; Aono, Tetsuya  
 PATENT ASSIGNEE(S): Takeda Chemical Industries Ltd, Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 31 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 08225574	A	19960903	JP 1995-330939	19951220 <--
PRIORITY APPLN. INFO.:			JP 1995-330939	A 19951220
			JP 1994-317938	19941221

OTHER SOURCE(S): MARPAT 125:329472  
 GI For diagram(s), see printed CA Issue.  
 AB The title compds. [I; ring A = (un)substituted five membered ring; Z = (un)substituted bivalent aliphatic chain consisting of a series of <5 atoms and optionally interrupted by one hetero atom in the chain; B = (un)substituted 5- or 6-membered heterocyclyl or carbocyclyl; the substituent of B is preferably CONHCH(CO<sub>2</sub>R<sub>3</sub>)(CH<sub>2</sub>)<sub>p</sub>WR<sub>4</sub>; wherein p = 1-4; W = bond, O, NHCONH, NR, NRCO, CONR, NHSO<sub>2</sub>; wherein R = H, C1-4 hydrocarbyl; CO<sub>2</sub>R<sub>3</sub> = optionally esterified CO<sub>2</sub>H; R<sub>4</sub> = (un)substituted chain or cyclic group; or Z = (CR<sub>1</sub>R<sub>2</sub>)<sub>n</sub>-Z<sub>1</sub>; wherein R<sub>1</sub>, R<sub>2</sub> = H, lower alkyl; Z<sub>1</sub> = bond, O, NH; n = 1-5] or salts thereof, which are useful for treating infections of

protozoa, particularly coccidium and drug-resistant malaria, are prepared Thus, 4-[2-(2,4-diamino-7H-pyrrolo[2,3-d]pyrimidin-5-yl)ethyl]benzoic acid ditrifluoroacetate was condensed with Me O-(4-methoxycarbonylbenzyl)-L-serinate hydrochloride using di-Et cyanophosphate and Et3N in DMF at room temperature for 1 h to give 66% Me N-[4-[2-(2,4-diamino-7H-pyrrolo[2,3-d]pyrimidin-5-yl)ethyl]benzoyl]-O-O-(4-methoxycarbonylbenzyl)-L-serinate, which was saponified with a mixture of 1 h aqueous NaOH and MeOH at room temperature for

5 h and neutralized with dilute HCl to give 84% N-[4-[2-(2,4-diamino-7H-pyrrolo[2,3-d]pyrimidin-5-yl)ethyl]benzoyl]-O-O-(4-methoxycarbonylbenzyl)-L-serine. These compds. I inhibited dihydrofolic acid reductase of malaria protozoa Plasmodium falciparum with IC50 of 0.8-62 nM. The title compound (II; R5 = Q) showed ED50 of 0.17 and 0.11 nM for inhibiting the proliferation of wild type-malaria protozoa P. falciparum 3D7 and cycloquanyl-resistant P. falciparum FCR3, resp. Capsule, tablet, and vial formulations containing II (R5 = Q1) were prepared

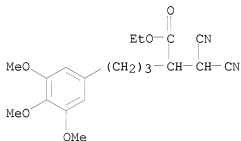
IT 182961-44-0P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation of ring-fused pyrimidine-containing amino acid derivs. as antiprotozoan agents)

RN 182961-44-0 CAPLUS

CN Benzenepentanoic acid,  $\alpha$ -(dicyanomethyl)-3,4,5-trimethoxy-, ethyl ester (CA INDEX NAME)



L4 ANSWER 21 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1996:365832 CAPLUS

DOCUMENT NUMBER: 125:86845

TITLE: Cyclopropenation and Related Reactions of Ruthenium Vinylidene Complexes

AUTHOR(S): Ting, Pei-Chen; Lin, Ying-Chih; Lee, Gene-Hsiang; Cheng, Ming-Chu; Wang, Yu

CORPORATE SOURCE: Department of Chemistry, National Taiwan University, Taipei, 106, Taiwan

SOURCE: Journal of the American Chemical Society (1996), 118(27), 6433-6444

CODEN: JACSAT; ISSN: 0002-7863

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Facile deprotonation of a number of cationic ruthenium vinylidene complexes, followed by cyclopropenation, is accomplished in acetone. The

deprotonation of  $[\text{Ru}]:\text{C}(\text{Ph})\text{CH}_2\text{R}^+$ , ( $[\text{Ru}] = (\eta^5\text{-C}_5\text{H}_5)(\text{PPh}_3)_2\text{Ru}$  through out this abstract) by  $n\text{-Bu}_4\text{NOH}$  induces a novel cyclization reaction and yields the neutral cyclopropenyl complexes [cyclic]  $[\text{Ru}]:\text{C}(\text{Ph})\text{CHR}$  (3b,  $\text{R} = \text{CN}$ ; 3c,  $\text{R} = \text{Ph}$ ; 3d,  $\text{R} = \text{CH}:\text{CH}_2$ ; 3e,  $\text{R} = \text{CH}:\text{CMe}_2$ ). Cyclic complex  $[\text{Ru}]:\text{C}(\text{C}_6\text{H}_9)\text{CHCN}^+$  is similarly prepared. Protonation of 3b-3e regenerates the corresponding vinylidene complexes. Deprotonation of  $[\text{Ru}]:\text{C}(\text{Ph})\text{CH}_2\text{COOMe}^+$  by  $n\text{-Bu}_4\text{NOH}$  induces a different type of cyclization and yields the neutral furan complex [cyclic]  $[\text{Ru}]:\text{C}(\text{Ph})\text{CH}:\text{C}(\text{O})\text{OMe}$  (4h). The cyclopropenyl complex containing a methoxy substituent cannot be prepared from  $[\text{Ru}]:\text{C}(\text{Ph})\text{CH}_2\text{OCH}_3^+$  (2i), but  $\text{F}^-$  of  $n\text{-Bu}_4\text{NF}$  attacks the  $\text{C}\alpha$  of 2i to produce the unstable vinyl complex  $[\text{Ru}]\text{C}(\text{F}):\text{C}(\text{Ph})\text{CH}_2\text{OCH}_3$ . Cyclic complex  $[\text{Ru}]:\text{C}(\text{Ph})\text{C}(\text{CN})\text{OCH}_3$  (9b) was indirectly prepared from the addition of TCNQ to 3b, giving [cyclic]  $[\text{Ru}]:\text{C}(\text{Ph})\text{CH}(\text{CN})\text{TCNQ}$  (6b) followed by methanolysis. Unlike 3, complex 9b is not converted to vinylidene complex, instead, removal of the methoxy substituent by acid gives the cationic cyclopropenyl complex  $[\text{Ru}]:\text{C}(\text{Ph})\text{C}(\text{CN})^+$ . Cyclic complex  $[\text{Ru}]:\text{C}(\text{Ph})\text{C}(\text{COOMe})^+$  is similarly prepared from 4h via a TCNQ complex followed by a methoxy-substituted complex. In the presence of allyl iodide, opening of the three-membered ring of 3b, followed by a subsequent oxidative coupling reaction, gives a dimeric dicationic product  $\{[\text{Ru}]:\text{C}(\text{Ph})\text{-CHCN}\}_2^{2+}$  (11). Proton abstraction of 11 by  $n\text{-Bu}_4\text{NOH}$  gives the biscyclopropenyl complex  $\{[\text{Ru}]:\text{C}(\text{Ph})\text{CCN}\}_2$ . Mol. structures of complexes 3b, 4h, 6b, 9b, 11, and [cyclic]  $[\text{Ru}]:\text{C}(\text{Ph})\text{C}(\text{CPh}_3)\text{CN}$  have been confirmed by x-ray diffraction anal.

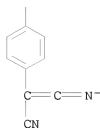
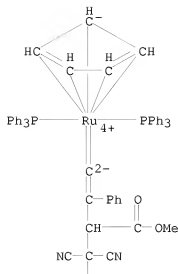
IT 178687-62-2P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(cyclopropenation and related reactions of ruthenium vinylidene complexes)

RN 178687-62-2 CAPLUS

CN Ruthenium, ( $\eta^5\text{-2,4-cyclopentadien-1-yl}$ )[4,4-dicyano-4-[4-(cyanoiminatoethenyl)phenyl]-3-(methoxycarbonyl)-2-phenyl-1-butenylidene]bis(triphenylphosphine)- (9CI) (CA INDEX NAME)



L4 ANSWER 22 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1996:6671 CAPLUS

DOCUMENT NUMBER: 124:177091

TITLE: Novel gem-dinitrile functionalized polyesters and polyamides from malononitrile; potential piezoelectric materials

AUTHOR(S): Steadman, Scott; Parrish, Dennis A.; Mathias, Lon J.

CORPORATE SOURCE: Department of Polymer Science, University of Southern

Mississippi, Hattiesburg, MS, 39406-0076, USA

SOURCE: Polymer Preprints (American Chemical Society, Division

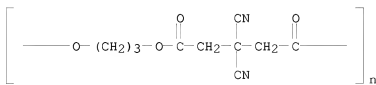
of Polymer Chemistry) (1995), 36(2), 320-1

CODEN: ACPPAY; ISSN: 0032-3934

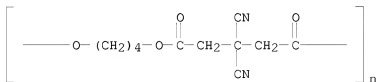
PUBLISHER: American Chemical Society, Division of Polymer Chemistry

DOCUMENT TYPE: Journal  
 LANGUAGE: English

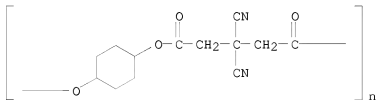
- AB Novel polyamides and polyesters in which the dinitrile group can potentially align in the same direction as the dipole of the carbonyl groups were synthesized via step growth dialkylation of malononitrile. Diamide and diester monomers facilitated polymerization by the attachment of chlorine to an activated position ( $\alpha$  to carbonyl). The polymers, having mol. weight 5000-8000, were characterized by NMR, viscosity and thermal anal.
- IT 169893-85-0P 174297-80-4P 174297-82-6P  
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of gem-dinitrile functionalized polyesters and polyamides from malononitrile as potential piezoelec. materials)
- RN 169893-85-0 CAPLUS
- CN Poly[oxy-1,3-propanediyl oxy(3,3-dicyano-1,5-dioxo-1,5-pentanediy)] (9CI)  
 (CA INDEX NAME)



- RN 174297-80-4 CAPLUS
- CN Poly[oxy-1,4-butanediyl oxy(3,3-dicyano-1,5-dioxo-1,5-pentanediy)] (9CI)  
 (CA INDEX NAME)



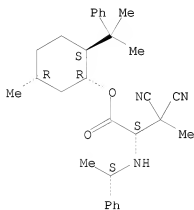
- RN 174297-82-6 CAPLUS
- CN Poly[oxy-1,4-cyclohexanediyloxy(3,3-dicyano-1,5-dioxo-1,5-pentanediy)] (9CI)  
 (CA INDEX NAME)



L4 ANSWER 23 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1995:900662 CAPLUS

DOCUMENT NUMBER: 124:116317  
 TITLE: Lanthanum isopropoxide catalyzed addition of activated nucleophiles to imines  
 AUTHOR(S): Yamamoto, Yoshinori; Fukui, Hiroyuki; Honda, Yoshihiro  
 CORPORATE SOURCE: Dept. Chem., Tohoku Univ., Sendai, 980-77, Japan  
 SOURCE: Applied Organometallic Chemistry (1995), 9(5 & 6), 467-71  
 CODEN: AOCHEX; ISSN: 0268-2605  
 PUBLISHER: Wiley  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 124:116317  
 AB The addition of certain activated nucleophiles to activated imines is catalyzed by lanthanum isopropoxide. As activated nucleophiles, methylmalonitrile and Me 2-cyanopropanoate can be utilized. Imines having an electron-withdrawing group either at the carbon or at the nitrogen atom of the C:N double bond can be used: for example N-toluenesulfonylimines, N-(4-methoxycarbonylphenyl)imines and  $\alpha$ -imino esters.  
 IT 155751-02-3P  
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (lanthanum isopropoxide catalyzed addition of activated nucleophiles to imines)  
 RN 155751-02-3 CAPLUS  
 CN Butanoic acid, 3,3-dicyano-2-[(1-phenylethyl)amino]-, 5-methyl-2-(1-methyl-1-phenylethyl)cyclohexyl ester, [1R-[1 $\alpha$ (S\*)(S\*),2 $\beta$ ,5 $\alpha$ ]]-(9CI) (CA INDEX NAME)

Absolute stereochemistry.

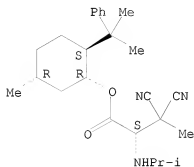


IT 155696-71-2P 155696-72-3P 172880-55-6P  
 172880-56-7P 173006-24-1P 173006-25-2P  
 173006-26-3P  
 RL: SPN (Synthetic preparation); PREP (Preparation) (lanthanum isopropoxide catalyzed addition of activated nucleophiles to imines)  
 RN 155696-71-2 CAPLUS  
 CN Butanoic acid, 3,3-dicyano-2-[(1-methylethyl)amino]-, 5-methyl-2-(1-methyl-1-phenylethyl)cyclohexyl ester, [1R-[1 $\alpha$ (S\*),2 $\beta$ ,5 $\alpha$ ]]-

10/923,271

(9CI) (CA INDEX NAME)

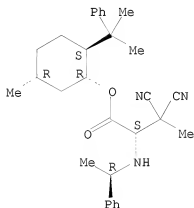
Absolute stereochemistry.



RN 155696-72-3 CAPLUS

CN Butanoic acid, 3,3-dicyano-2-[(1-phenylethyl)amino]-, 5-methyl-2-(1-methyl-1-phenylethyl)cyclohexyl ester, [1R-[1α[S\*(R\*)],2β,5α]]- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

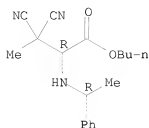


RN 172880-55-6 CAPLUS

CN Butanoic acid, 3,3-dicyano-2-[(1-phenylethyl)amino]-, butyl ester, (R\*,R\*)- (9CI) (CA INDEX NAME)

Relative stereochemistry.

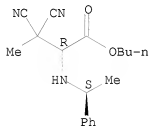
10/923,271



RN 172880-56-7 CAPLUS

CN Butanoic acid, 3,3-dicyano-2-[(1-phenylethyl)amino]-, butyl ester,  
(R\*, S\*)- (9CI) (CA INDEX NAME)

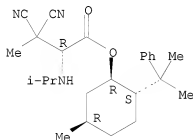
Relative stereochemistry.



RN 173006-24-1 CAPLUS

CN Butanoic acid, 3,3-dicyano-2-[(1-methylethyl)amino]-, 5-methyl-2-(1-methyl-1-phenylethyl)cyclohexyl ester, [1R-[1α(R\*), 2β, 5α]]-  
(9CI) (CA INDEX NAME)

Absolute stereochemistry.

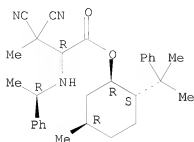


RN 173006-25-2 CAPLUS

CN Butanoic acid, 3,3-dicyano-2-[(1-phenylethyl)amino]-, 5-methyl-2-(1-methyl-1-phenylethyl)cyclohexyl ester, [1R-[1α(R\*), 2β, 5α]]-  
(9CI) (CA INDEX NAME)

Absolute stereochemistry.

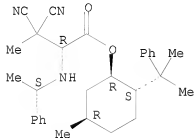




RN 173006-26-3 CAPLUS

CN Butanoic acid, 3,3-dicyano-2-[(1-phenylethyl)aminol]-, 5-methyl-2-(1-methyl-1-phenylethyl)cyclohexyl ester, [1R-[1 $\alpha$ [R\*(S\*)],2 $\beta$ ,5 $\alpha$ ]]-(9CI) (CA INDEX NAME)

Absolute stereochemistry.



L4 ANSWER 24 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1995:845626 CAPLUS

DOCUMENT NUMBER: 124:86769

TITLE: Novel three-component reaction of 1,1-dicyano-2-(trifluoromethyl)ethylenes with primary arylamines and ketones

AUTHOR(S): Tyutin, V. Yu.; Chkanikov, N. D.; Nesterov, V. N.; Antipin, M. Yu.; Struchkov, Yu. T.; Kolomiets, A. F.; Fokin, A. V.

CORPORATE SOURCE: A. N. Nesmeyanov Inst. Organoelem. Compd., Russ. Acad. Sci., Moscow, 117813, Russia

SOURCE: Izvestiya Akademii Nauk, Seriya Khimicheskaya (1993), (3), 552-9

CODEN: IASKEA

PUBLISHER: Nauka

DOCUMENT TYPE: Journal

LANGUAGE: Russian

OTHER SOURCE(S): CASREACT 124:86769

AB 1,1-Dicyano-2,2-bis(trifluoromethyl)ethylene and 3,3-dicyano-2-(trifluoromethyl)acrylates react with primary arylamines in the presence of ketones to form 1,1-aryl-1,4-dihydropyridine derivs. under mild conditions. In this three-component reaction Schiff bases are formed as intermediates. 1,4-Dihydropyridines derivs., which are the products of three-component heterocyclization, were also obtained by interaction of

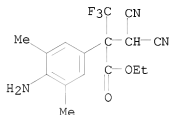
the corresponding Schiff bases with 1,1-dicyano-2-(trifluoromethyl)ethylenes.

IT 134641-39-7P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 134641-39-7 CAPLUS

CN Benzeneacetic acid, 4-amino- $\alpha$ -(dicyanomethyl)-3,5-dimethyl- $\alpha$ -(trifluoromethyl)-, ethyl ester (CA INDEX NAME)



L4 ANSWER 25 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1995:695727 CAPLUS

DOCUMENT NUMBER: 123:286966

TITLE: Novel gem-dinitrile functionalized polyesters and polyamides from malononitrile; potential piezoelectric materials

AUTHOR(S): Mathias, Lon J.; Parrish, Dennis A.; Steadman, Scott

CORPORATE SOURCE: Department Polymer Science, University Southern Mississippi, Hattiesburg, MS, 39406-0076, USA

SOURCE: Polymer Preprints (American Chemical Society, Division of Polymer Chemistry) (1994), 35(2), 659-60  
CODEN: ACPPAY; ISSN: 0032-3934

PUBLISHER: American Chemical Society, Division of Polymer Chemistry

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The initial success is described in obtaining a polyester and polyamide in which the dinitrile group net dipole can potentially align in the same direction as the carbonyl groups.

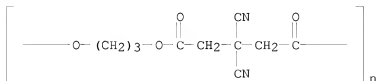
IT 169893-85-0P

RL: SPN (Synthetic preparation); PREP (Preparation)

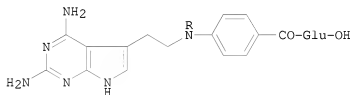
(preparation of gem-dinitrile functionalized polyesters and polyamides from malononitrile as potential piezoelec. materials)

RN 169893-85-0 CAPLUS

CN Poly[oxy-1,3-propanediyloxy(3,3-dicyano-1,5-dioxo-1,5-pentanediy)] (9CI)  
(CA INDEX NAME)



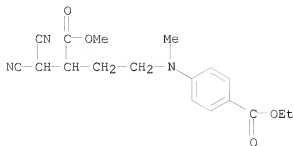
L4 ANSWER 26 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1995:440268 CAPLUS  
 DOCUMENT NUMBER: 123:112653  
 TITLE: Synthesis and antitumor activity of  
 pyrrolo[2,3-d]pyrimidine antifolates with a bridge  
 chain containing a nitrogen atom  
 AUTHOR(S): Aso, Kazuyoshi; Hitaka, Takenori; Yukishige, Koichi;  
 Ootsu, Koichiro; Akimoto, Hiroshi  
 CORPORATE SOURCE: Pharmaceutical Res. Div., Takeda Chem. Industries,  
 Ltd., Osaka, 532, Japan  
 SOURCE: Chemical & Pharmaceutical Bulletin (1995),  
 43(2), 256-61  
 CODEN: CPBTAL; ISSN: 0009-2363  
 PUBLISHER: Pharmaceutical Society of Japan  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 123:112653  
 GI



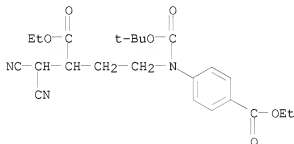
AB Novel pyrrolo[2,3-d]pyrimidine antifolates I (R = H, Me) with a nitrogen atom in the bridge chain between the 2,4-diaminopyrrolo[2,3-d]pyrimidine and phenylene rings were designed and efficiently synthesized. I exhibited more potent inhibitory activities than methotrexate (MTX) against the proliferation of human epidermoid carcinoma KB cells and human non-small cell lung carcinoma A549 cells despite their modest dihydrofolate reductase (DHFR)-inhibitory potency.

IT 133719-38-7P 133719-41-2P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (preparation and antitumor activity of pyrrolo[2,3-d]pyrimidine antifolates with nitrogen-containing bridge chains)

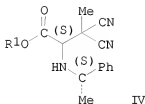
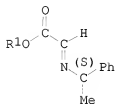
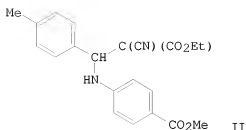
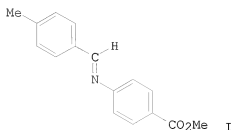
RN 133719-38-7 CAPLUS  
 CN Benzoic acid, 4-[[4,4-dicyano-3-(methoxycarbonyl)butyl]methylamino]-, ethyl ester (CA INDEX NAME)



RN 133719-41-2 CAPLUS  
 CN Benzoic acid, 4-[[[4,4-dicyano-3-(ethoxycarbonyl)butyl][(1,1-dimethylethoxy)carbonyl]amino]-, ethyl ester (CA INDEX NAME)



L4 ANSWER 27 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1994:434408 CAPLUS  
 DOCUMENT NUMBER: 121:34408  
 ORIGINAL REFERENCE NO.: 121:6341a,6342a  
 TITLE: Transition metal catalyzed addition of certain nucleophiles to imines  
 AUTHOR(S): Yamamoto, Yoshinori; Kubota, Yasufumi; Honda, Yoshihiro; Fukui, Hiroyuki; Asao, Naoki; Nemoto, Hisao  
 CORPORATE SOURCE: Faculty of Science, Tohoku University, Sendai, 980, Japan  
 SOURCE: Journal of the American Chemical Society (1994), 116(7), 3161-2  
 CODEN: JACSAT; ISSN: 0002-7863  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 121:34408  
 GI



AB Imines react with certain nucleophiles in the presence of catalytic amts. of transition metal complexes to give alkylation products in good yield. Thus, imine (I) was treated with  $\text{CH}(\text{CN})_2(\text{CO}_2\text{Et})$  in the presence of  $\text{RhHCO}(\text{PPh}_3)_3$  in THF solvent to give alkylation product II in 75% yield. A significantly high diastereomeric excess was accomplished by using III [ $\text{R}_1 = (-)-8\text{-phenylmenthyl}$ ] in which a chiral auxiliary exists at the ester unit. The  $\text{Ls}(\text{O-isopropyl})$  catalyzed reaction of III with  $\text{CH}(\text{CN})_2\text{Me}$  in THF at room temperature gave IV ( $\text{R}_1$  as above) as the predominant diastereoisomer in a 90:10 ratio; x-ray anal. indicate that the  $\alpha$ -carbon to the amino group possesses the *S* configuration.

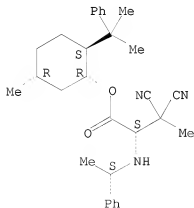
IT 155751-02-3P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of, absolute configuration of)

RN 155751-02-3 CAPLUS

CN Butanoic acid, 3,3-dicyano-2-[(1-phenylethyl)amino]-, 5-methyl-2-[(1-methyl-1-phenylethyl)cyclohexyl ester, [1R-[1 $\alpha$ [S\*(S\*)],2 $\beta$ ,5 $\alpha$ ]]-(9CI) (CA INDEX NAME)

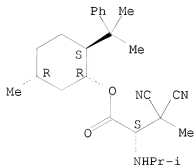
Absolute stereochemistry.



10/923,271

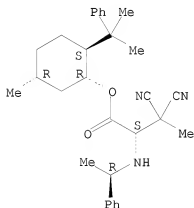
IT 155696-71-2P 155696-72-3P  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of, stereoselective)  
RN 155696-71-2 CAPLUS  
CN Butanoic acid, 3,3-dicyano-2-[(1-methylethyl)amino]-, 5-methyl-2-(1-methyl-1-phenylethyl)cyclohexyl ester, [1R-[1 $\alpha$ (S\*),2 $\beta$ ,5 $\alpha$ ]]-(9CI) (CA INDEX NAME)

Absolute stereochemistry.



RN 155696-72-3 CAPLUS  
CN Butanoic acid, 3,3-dicyano-2-[(1-phenylethyl)amino]-, 5-methyl-2-(1-methyl-1-phenylethyl)cyclohexyl ester, [1R-[1 $\alpha$ (S\*(R\*)),2 $\beta$ ,5 $\alpha$ ]]-(9CI) (CA INDEX NAME)

Absolute stereochemistry.



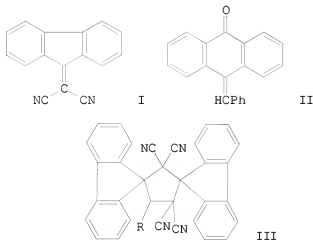
L4 ANSWER 28 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
ACCESSION NUMBER: 1994:269808 CAPLUS  
DOCUMENT NUMBER: 120:269808  
ORIGINAL REFERENCE NO.: 120:47779a,47782a  
TITLE: Wittig reactions of a fluoren-9-ylidene and an anthrone-10-arylidene  
AUTHOR(S): Ganoub, Neven A. F.

CORPORATE SOURCE:  
SOURCE:

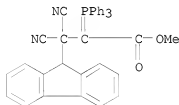
Dep. Pesticide Chem., Natl. Res. Cent., Cairo, Egypt  
Phosphorus, Sulfur and Silicon and the Related  
Elements (1993), 81(1-4), 125-31  
CODEN: PSSLEC; ISSN: 1042-6507

DOCUMENT TYPE:  
LANGUAGE:  
OTHER SOURCE(S):  
GI

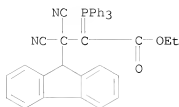
Journal  
English  
CASREACT 120:269808



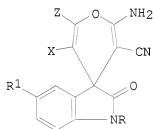
- AB The Wittig reactions of fluorene-9-ylidenemalonitrile (I) and 10-benzylideneanthrone (II) with phosphonium ylides  $\text{Ph}_3\text{P}^+\text{CH}^-\text{CO}_2\text{R}$  ( $\text{R} = \text{Me}, \text{Et}$ ) have been investigated. In both cases, unusual reaction products, e.g., bis(9-fluorenyl)cyclopentane III (from I), were isolated and identified on the basis of elemental analyses and spectral studies.
- IT 154496-99-8P 154497-00-4P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(preparation and thermal decomposition of)
- RN 154496-99-8 CAPLUS
- CN 9H-Fluorene-9-propanoic acid,  $\beta,\beta$ -dicyano- $\alpha$ -(triphenylphosphoranylidene)-, methyl ester (CA INDEX NAME)



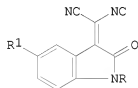
- RN 154497-00-4 CAPLUS
- CN 9H-Fluorene-9-propanoic acid,  $\beta,\beta$ -dicyano- $\alpha$ -(triphenylphosphoranylidene)-, ethyl ester (CA INDEX NAME)



L4 ANSWER 29 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1994:244629 CAPLUS  
 DOCUMENT NUMBER: 120:244629  
 ORIGINAL REFERENCE NO.: 120:43353a, 43356a  
 TITLE: Synthesis of spiro indolin-2-one derivatives  
 AUTHOR(S): El-Ahl, Abdel Aziz S.; Afeefy, Hussein; Metwally, Mohamed Abbas  
 CORPORATE SOURCE: Fac. Sci., Mansoura Univ., Mansoura, Egypt  
 SOURCE: Journal of Chemical Research, Synopses (1994), (1), 14-15  
 CODEN: JRPSDC; ISSN: 0308-2342  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 120:244629  
 GI



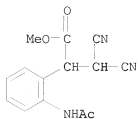
I



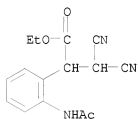
II

AB Title compds. I were prepared by heating dicyanomethyleneindolinones II (R = H, R1 = H, Me; R = Me, Ac, R1 = H) with active methylene compds., XCH2COZ (X = Ac, Z = OEt, Me; X = cyano, Z = Ph).  
 IT 154379-70-1P 154379-71-2P  
 RL: SPN (Synthetic preparation); PREP (Preparation) (preparation of)  
 RN 154379-70-1 CAPLUS  
 CN Benzeneacetic acid, 2-(acetylamino)- $\alpha$ -(dicyanomethyl)-, methyl ester (CA INDEX NAME)





RN 154379-71-2 CAPLUS

CN Benzeneacetic acid, 2-(acetylamino)-α-(dicyanomethyl)-, ethyl ester  
(CA INDEX NAME)

L4 ANSWER 30 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1993:213100 CAPLUS

DOCUMENT NUMBER: 118:213100

ORIGINAL REFERENCE NO.: 118:36739a,36742a

TITLE: Preparation of tricyclic fused pyrimidine compounds

INVENTOR(S): Akimoto, Hiroshi; Otsu, Koichiro; Miwa, Tetsuo

PATENT ASSIGNEE(S): Takeda Chemical Industries, Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 25 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

## PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 04211063	A	19920803	JP 1991-65613	19910305 <--
			JP 1990-54620	A1 19900305

PRIORITY APPLN. INFO.:

OTHER SOURCE(S): MARPAT 118:213100

GI For diagram(s), see printed CA Issue.

AB The title comps. [I; Q1 = H, halo, radical linked through C, N, O, or S; one of Q2 and Q3 = N, the other = N, CH; Y = N, CR1 (wherein R1 = H, hydrocarbyl), methylidyne; Z = C2-5 bivalent radical containing optional substituents; ring A1, A2 = (substituted) 5-7-membered ring; B = (substituted) cyclic radical, etc.], useful as antitumor agents with high selectivity, are prepared Cyclocondensation of 1.181 g ester II (preparation given) with 314 mg guanidine HCl and Me3COK in Me3COH gave 1.02 g pyrrolopyrimidine III, which (1.010 g) was treated with borane-THF complex in THF at 0° and then at 50°, the solution cooled and stirred with HOAc-MeOH at room temperature to give 542 mg IV. The preferred doses of I

are 2.0-500 mg/kg-day orally and 1.0-200 mg/kg injection.

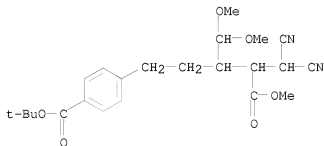
IT 147239-87-0P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation and reaction of, in preparation of antitumor agent)

RN 147239-87-0 CAPLUS

CN Benzenepentanoic acid,  $\alpha$ -(dicyanomethyl)- $\beta$ -(dimethoxymethyl)-4-[(1,1-dimethylethoxy)carbonyl]-, methyl ester (CA INDEX NAME)



L4 ANSWER 31 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1993:124655 CAPLUS

DOCUMENT NUMBER: 118:124655

ORIGINAL REFERENCE NO.: 118:21621a, 21624a

TITLE: Wittig reaction of 1-(dicyanomethylene)acenaphthen-2-one

AUTHOR(S): Abdou, Wafaa M.; Ganoub, Neven A. F.

CORPORATE SOURCE: Natl. Res. Cent., Cairo, Egypt

SOURCE: Heteroatom Chemistry (1992), 3(2), 133-7

CODEN: HETCE8; ISSN: 1042-7163

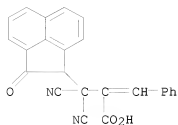
DOCUMENT TYPE: Journal

LANGUAGE: English

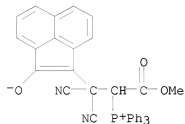
OTHER SOURCE(S): CASREACT 118:124655

GI





IT 145882-82-2P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
 (Reactant or reagent)  
 (preparation, Wittig olefination, and thermal intramol. cyclocondensation  
 of)  
 RN 145882-82-2 CAPLUS  
 CN Phosphonium, [2,2-dicyano-2-(2-hydroxy-1-acenaphthylene)-1-  
 (methoxycarbonyl)ethyl]triphenyl-, inner salt (CA INDEX NAME)



L4 ANSWER 32 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1993:29863 CAPLUS  
 DOCUMENT NUMBER: 118:29863  
 ORIGINAL REFERENCE NO.: 118:5361a,5364a  
 TITLE: Silver halide photographic material containing a  
 compound which releases photographically useful  
 species upon development  
 INVENTOR(S): Asatake, Atsushi  
 PATENT ASSIGNEE(S): Konica Co., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 19 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 04177243	A	19920624	JP 1990-305540	19901110 <--
PRIORITY APPLN. INFO.:			JP 1990-305540	19901110

AB The photog. material contains a compound CRR1R2C(R3R4)mZ(Z1)nPUG (R =  
 leaving group released by nucleophilic substitution; R1,R2,R3,R4 = H,

aliphatic, aromatic, heterocyclic or electron-attracting group; Z = electron-attracting group; Z1 = timing group to be subjected to break and release PUG: PUG = photog. useful group; m,n = 0, 1). The photog. material has good storage stability, while upon development, it releases the PUGs at a proper reaction rate even in developer solution of relatively low pH.

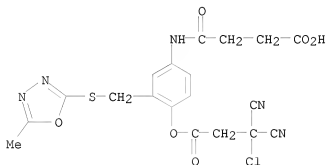
IT 144896-71-9 145059-42-3

RL: USES (Uses)

(photog. useful group-releasing, in processing)

RN 144896-71-9 CAPLUS

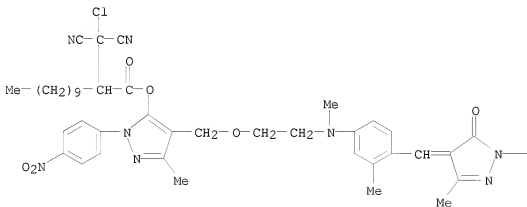
CN Butanoic acid, 4-[[4-(3-chloro-3,3-dicyano-1-oxopropoxy)-3-[[5-methyl-1,3,4-oxadiazol-2-yl]thio]methyl]phenyl]amino]-4-oxo- (CA INDEX NAME)



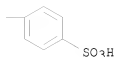
RN 145059-42-3 CAPLUS

CN Dodecanoic acid, 2-(chlorodicyanomethyl)-, 4-[[2-[[4-[[1,5-dihydro-3-methyl-5-oxo-1-(4-sulfophenyl)-4H-pyrazol-4-ylidene]methyl]-3-methylphenyl]methylamino]ethoxy]methyl]-3-methyl-1-(4-nitrophenyl)-1H-pyrazol-5-yl ester, monopotassium salt (9CI) (CA INDEX NAME)

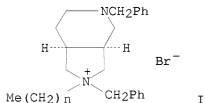
PAGE 1-A



● K

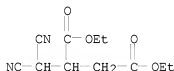


L4 ANSWER 33 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1992:174018 CAPLUS  
 DOCUMENT NUMBER: 116:174018  
 ORIGINAL REFERENCE NO.: 116:29451a,29454a  
 TITLE: Synthesis and structure-antimicrobial activity relationships of quaternary ammonium derivatives of perhydropyrrolo-[3,4-c]pyridine  
 AUTHOR(S): Altomare, C.; Carotti, A.; Casini, G.; Cellamare, S.; Ferappi, M.; Vitali, C.  
 CORPORATE SOURCE: Dip. Farm. Chim., Univ. Bari, Bari, I-70125, Italy  
 SOURCE: Arzneimittel-Forschung (1992), 42(2), 152-5  
 CODEN: ARZNAD; ISSN: 0004-4172  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 116:174018  
 GI

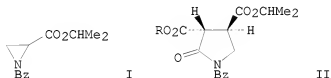


AB A homologous series of perhydropyrrolo[3,4-c]pyridine quaternary ammonium derivs. I (n = 5, 7, 9-13,15) was synthesized from EtO<sub>2</sub>CCH<sub>2</sub>CH(CO<sub>2</sub>Et)CH(CN)<sub>2</sub> and tested for in vitro antibacterial activity against different gram-pos. and gram-neg. bacteria. All I were more potent than the reference compound, benzalkonium chloride. Antibacterial activity, expressed as log 1/MIC, was linearly related to lipophilicity up to C13-C14 homologs, where a break in the linear relationship was observed

IT 82584-86-9  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (hydrogenation-intramol. cyclocondensation of)  
 RN 82584-86-9 CAPLUS  
 CN Butanedioic acid, (dicyanomethyl)-, diethyl ester (9CI) (CA INDEX NAME)

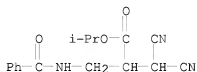


L4 ANSWER 34 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1992:83465 CAPLUS  
 DOCUMENT NUMBER: 116:83465  
 ORIGINAL REFERENCE NO.: 116:14203a,14206a  
 TITLE: The regioselectivity of the ring opening of  
 1-activated or nonactivated 2-alkoxycarbonyl or  
 2-cyanoaziridines by carbanions of the dicarbonyl  
 compounds  
 AUTHOR(S): Bouayad, Zoheir; Chanet-Ray, Josette; Ducher, S.;  
 Vessiere, Roger  
 CORPORATE SOURCE: Ec. Natl. Super. Chim. Clermont-Ferrand, Univ. Blaise  
 Pascal, Aubiere, 63177, Fr.  
 SOURCE: Journal of Heterocyclic Chemistry (1991),  
 28(7), 1757-67  
 CODEN: JHTCAD; ISSN: 0022-152X  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 GI

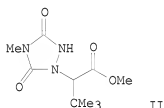


AB Aziridines, e.g. I, reacted with carbanions of dicarbonyl compds., e.g.  
 RO<sub>2</sub>CCH<sub>2</sub>CO<sub>2</sub>R (R = Me, Et, CHMe<sub>2</sub>), to give ring opened products and/or ring  
 enlarged products, e.g. (RO<sub>2</sub>C)CHCH<sub>2</sub>CH(NHBz)CO<sub>2</sub>CHMe<sub>2</sub>,  
 (RO<sub>2</sub>C)CHCH(CO<sub>2</sub>CHMe<sub>2</sub>)CH<sub>2</sub>NHBz, and pyrrole II. The regioselectivity  
 depends on several factors. The Ph group on C-3 favors C-3-N bond  
 cleavage, whereas C-2-N bond cleavage is predominant with C-3 substituted  
 or C-2-H aziridines. Cyanoaziridines are predominantly cleaved at C-3-N.  
 IT 138478-35-0P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)  
 RN 138478-35-0 CAPLUS

CN Propanoic acid, 2-[(benzoylamino)methyl]-3,3-dicyano-, 1-methylethyl ester  
(CA INDEX NAME)



L4 ANSWER 35 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1991:680104 CAPLUS  
 DOCUMENT NUMBER: 115:280104  
 ORIGINAL REFERENCE NO.: 115:47607a, 47610a  
 TITLE: E/Z isomerization, solvolysis, addition, and  
 cycloaddition reactions of (E)-tert-butylketene methyl  
 tert-butylidimethylsilyl acetal  
 AUTHOR(S): Adam, Waldemar; Wang, Xiaoheng  
 CORPORATE SOURCE: Inst. Org. Chem., Univ. Wuerzburg, Wuerzburg, D-8700,  
 Germany  
 SOURCE: Journal of Organic Chemistry (1991), 56(26),  
 7244-50  
 CODEN: JOCEAH; ISSN: 0022-3263  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 115:280104  
 GI



AB In the presence of catalytic amts. of CF<sub>3</sub>COMe or CF<sub>3</sub>COCF<sub>3</sub>, the silyl ketene acetal Me<sub>3</sub>CCH:C(OMe)OSiMe<sub>2</sub>CMe<sub>3</sub> (E-I) was isomerized into its Z isomer (Z/E ratio 90:10). For this novel E/Z isomerization a mechanism is proposed in which addition and reelimination of the fluoro ketone through a 1,4-dipolar intermediate operates. With the protic nucleophiles MeOH, CF<sub>3</sub>CH<sub>2</sub>OH, or PhOH, the ketene acetal E-I afforded the ortho esters Me<sub>3</sub>CCH<sub>2</sub>C(OMe)(OR)OSiMe<sub>2</sub>CMe<sub>3</sub> (R = Me, CF<sub>3</sub>CH<sub>2</sub>, Ph) as addition products, while AcOH, CF<sub>3</sub>CO<sub>2</sub>H, or H<sub>2</sub>O led to Me pivalate as the solvolysis product. This chemical is readily explained through protonation of the ketene acetal E-I to generate the corresponding carbenium ion. At low temperature the reaction with TCNE gave the silylketene imine as labile cycloadduct, which underwent desilylation on workup to give the TCNE-incorporated ester



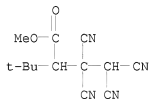
(NC)2CHC(CN)2CH(CMe2)C(O)OMe; the latter eliminated hydrogen cyanide at room temperature to give the ene ester. With MTAD the labile silyl ene product was obtained initially, which underwent silyl migration to give N-silylated urazole; final desilylation led to the stable urazole II. Also, for the ene reactions of TCNE and MTAD with the silyl ketene acetal E-I, intervention of a 1,4-dipolar intermediate is proposed.

IT 136911-64-3P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation and hydrogen cyanide elimination of)

RN 136911-64-3 CAPLUS

CN Butanoic acid, 3,3,4,4-tetracyano-2-(1,1-dimethylethyl)-, methyl ester (9CI) (CA INDEX NAME)

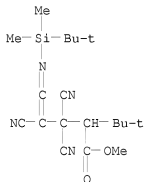


IT 136911-63-2P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 136911-63-2 CAPLUS

CN 4-Pentenoic acid, 3,3,4-tricyano-2-(1,1-dimethylethyl)-5-[[[1,1-dimethylethyl]dimethylsilylimino]-, methyl ester (CA INDEX NAME)



L4 ANSWER 36 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

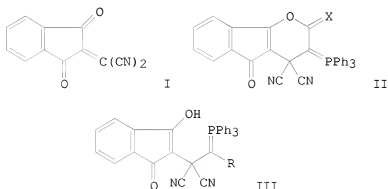
ACCESSION NUMBER: 1991:608100 CAPLUS

DOCUMENT NUMBER: 115:208100

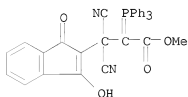
ORIGINAL REFERENCE NO.: 115:35517a,35520a

TITLE: Chemistry of phosphorus ylides. 10. Reaction with phosphacumulenes. IV. Synthesis of pyran, phosphoranylidene, oxaphosphorin and oxazaphosphorin from the reaction of 1,3-dioxo-A2,α-indanmalononitrile with phosphoranes and

iminophosphoranes  
 AUTHOR(S): Soliman, Fouad M.; Said, Medhat M.  
 CORPORATE SOURCE: Natl. Res. Cent., Cairo, Egypt  
 SOURCE: Phosphorus, Sulfur and Silicon and the Related  
 Elements (1991), 61(3-4), 335-40  
 CODEN: PSSLEC; ISSN: 1042-6507  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 115:208100  
 GI

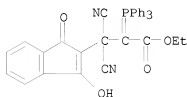


AB 1,3-Dioxo-Δ2,α-indanmalononitrile (I) reacts with the active  
 ketenylidene- and thioketenylidenetriphenylphosphoranes Ph3P:C:C:X (X = O,  
 S, resp.) to give the corresponding pyrans II (X = O, S). The reaction of  
 II with 4-O2NC6H4CHO proceeds according to the Wittig reaction to give the  
 resp. methyldene derivs. On the other hand, phosphoranylidenes III (R =  
 acyl, alkoxycarbonyl) were isolated from the reaction of stable  
 phosphoranes Ph3P:CHR with I. Moreover, an oxaphosphorin and  
 oxazaphosphorin were prepared from the reaction of I with the phosphorane  
 Ph3P:CPh2 and the iminophosphorane Ph3P:NCO2Et, resp.  
 IT 136829-50-0P 136848-91-4P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)  
 RN 136829-50-0 CAPLUS  
 CN 1H-Indene-2-propanoic acid, β,β-dicyano-3-hydroxy-1-oxo-α-  
 (triphenylphosphoranylidene)-, methyl ester (CA INDEX NAME)



RN 136848-91-4 CAPLUS  
 CN 1H-Indene-2-propanoic acid, β,β-dicyano-3-hydroxy-1-oxo-α-

(triphenylphosphoranylidene)-, ethyl ester (CA INDEX NAME)



L4 ANSWER 37 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1991:492003 CAPLUS

DOCUMENT NUMBER: 115:92003

ORIGINAL REFERENCE NO.: 115:15823a,15826a

TITLE: C-Alkylation of indoles with 1,1-bis(trifluoromethyl)-2,2-dicyanoethylene and 2-trifluoromethyl-3,3-dicyanoacrylic acid esters

AUTHOR(S): Chkanikov, N. D.; Komarov, K. V.; Tyutin, V. Yu.; Kolomiets, A. F.; Fokin, A. V.

CORPORATE SOURCE: Inst. Elementoorg. Soedin. im. Nesmeyanova, Moscow, USSR

SOURCE: Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1991), (5), 1193-5

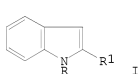
CODEN: IASKA6; ISSN: 0002-3353

DOCUMENT TYPE: Journal

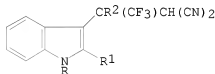
LANGUAGE: Russian

OTHER SOURCE(S): CASREACT 115:92003

GI



I



II

AB The indole derivs. I (R = H, Me; R1 = H, Me, Ph) were alkylated with (NC)2C:CR2CF3 (R2 = CF3, CO2Me, CO2Et) to give the corresponding dicyanoethyl derivs. II.

IT 135578-14-2P 135578-15-3P 135578-17-5P

135578-18-6P 135578-19-7P 135578-20-0P

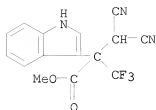
RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)

RN 135578-14-2 CAPLUS

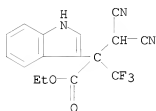
CN 1H-indole-3-acetic acid,  $\alpha$ -(dicyanomethyl)- $\alpha$ -(trifluoromethyl)-, methyl ester (CA INDEX NAME)

10/923,271



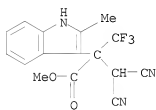
RN 135578-15-3 CAPLUS

CN 1H-Indole-3-acetic acid,  $\alpha$ -(dicyanomethyl)- $\alpha$ -(trifluoromethyl)-, ethyl ester (CA INDEX NAME)



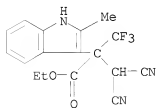
RN 135578-17-5 CAPLUS

CN 1H-Indole-3-acetic acid,  $\alpha$ -(dicyanomethyl)-2-methyl- $\alpha$ -(trifluoromethyl)-, methyl ester (CA INDEX NAME)



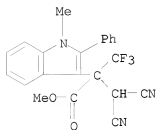
RN 135578-18-6 CAPLUS

CN 1H-Indole-3-acetic acid,  $\alpha$ -(dicyanomethyl)-2-methyl- $\alpha$ -(trifluoromethyl)-, ethyl ester (CA INDEX NAME)



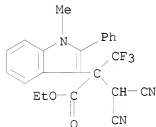
RN 135578-19-7 CAPLUS

CN 1H-Indole-3-acetic acid,  $\alpha$ -(dicyanomethyl)-1-methyl-2-phenyl- $\alpha$ -(trifluoromethyl)-, methyl ester (CA INDEX NAME)



RN 135578-20-0 CAPLUS

CN 1H-Indole-3-acetic acid,  $\alpha$ -(dicyanomethyl)-1-methyl-2-phenyl- $\alpha$ -(trifluoromethyl)-, ethyl ester (CA INDEX NAME)



L4 ANSWER 38 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1991:471633 CAPLUS

DOCUMENT NUMBER: 115:71633

ORIGINAL REFERENCE NO.: 115:12391a, 12394a

TITLE: Preparation of pyrrolopyrimidines as antitumor agents

INVENTOR(S): Akimoto, Hiroshi; Hitaka, Takenori

PATENT ASSIGNEE(S): Takeda Chemical Industries, Ltd., Japan

SOURCE: Eur. Pat. Appl., 27 pp.

CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 418924	A2	19910327	EP 1990-118202	19900921 <--
EP 418924	A3	19911023		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE				
JP 03173890	A	19910729	JP 1990-249615	19900918 <--
CA 2025830	A1	19910322	CA 1990-2025830	19900920 <--
US 5354754	A	19941011	US 1993-46917	19930414 <--
PRIORITY APPLN. INFO.:				
			JP 1989-245998	A 19890921
			US 1990-585950	B1 19900921

10/923,271

OTHER SOURCE(S): MARPAT 115:71633  
GI

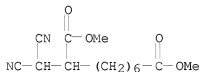
\* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT \*

AB Pyrrolopyrimidines [I; R1, R2 = H, ester residue; X = NH2, OH, SH; Y = H, OH; Z = (substituted) C2-4 divalent radical; Z1 = (substituted) divalent heterocycle residue, alkylene; dotted line indicates saturation or unsatn] are prepared Acetal II (1.32 g) (preparation given) was dissolved in CF3CO2H containing H2O with stirring at room temperature to give quant. salt III, which was dissolved with di-Et glutamate HCl in DMF and the solution was treated with 0.514 g H2NP(O)(OEt)2 and Et3N in DMF at room temperature to give 1.11 g diester IV (R1 = R2 = Et) (V). Saponification of 1.05 g V in THF gave 0.826 g acid IV (R1 = R2 = H), which showed IC50 of 0.00043 µg/mL against human epidermoid carcinoma KB cells.

IT 135110-11-1P 135111-93-2P  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (preparation and reaction of, in preparation of antitumor agent)

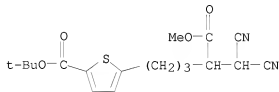
RN 135110-11-1 CAPLUS

CN Nonanedioic acid, 2-(dicyanomethyl)-, dimethyl ester (9CI) (CA INDEX NAME)



RN 135111-93-2 CAPLUS

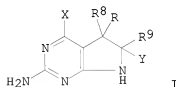
CN 2-Thiophenepentanoic acid, α-(dicyanomethyl)-5-[(1,1-dimethylethoxy)carbonyl]-, methyl ester (CA INDEX NAME)



L4 ANSWER 39 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
ACCESSION NUMBER: 1991:429914 CAPLUS  
DOCUMENT NUMBER: 115:29914  
ORIGINAL REFERENCE NO.: 115:5281a,5284a

TITLE: Preparation of N-[(pyrrolopyrimidinylethyl)amino]benzoylglutamates and analogs as antitumor agents  
 INVENTOR(S): Akimoto, Hiroshi; Hitaka, Takenori; Miwa, Tetsuo  
 PATENT ASSIGNEE(S): Takeda Chemical Industries, Ltd., Japan  
 SOURCE: Eur. Pat. Appl., 51 pp.  
 CODEN: EPXXDW  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 400562	A1	19901205	EP 1990-110131	19900529 <--
EP 400562	B1	19960821		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE				
JP 04009382	A	19920114	JP 1990-136345	19900525 <--
JP 3015957	B2	20000306		
CA 2017604	A1	19901129	CA 1990-2017604	19900528 <--
AT 141603	T	19960915	AT 1990-110131	19900529 <--
PRIORITY APPLN. INFO.:			JP 1989-135642	A 19890529
			JP 1989-246209	A 19890920
			JP 1990-93370	A 19900409
OTHER SOURCE(S):		MARPAT 115:29914		
GI				



AB The title compds. [I; R = (CR1R2)iZ1(CR4R5)jZ2CONHCH(CO2R6)CH2CH2CO2R7; R1, R2, R4, R5 = H, hydrocarbaryl, bond; R6, R7 = H, alkyl, (un)substituted Ph, PhCH2; R8, R9 = H; R8R9 = bond; X = NH2, OH, SH; Y = H, OH; Z1 = O, SOn, (alkyl)imino, etc.; Z2 = (un)substituted alkylene, divalent cyclic group; i, j = 0-3 (i + j = 1-3); n = 0-2] were prepared. Thus, 4-(EtO2C)C6H4NMeCH2CH2CH(CO2Me)CH(CN)2 (preparation given) was cyclocondensed with guanidine and the product reduced to give, as 1 of 2 products, anilinoethylpyrrolopyrimidine I [R = CH2CH2NMeC6H4(COR10)-4; R8R9 = bond, X = NH2, Y = H] (II; R10 = OEt) which was condensed with di-Et L-glutamate to give, after saponification, L-II [R10 = NHCH(CO2H)CH2CH2CO2H]. The latter

had

IC50 of 0.0013  $\mu$ M against human epidermoid carcinoma KB cell growth in vitro.

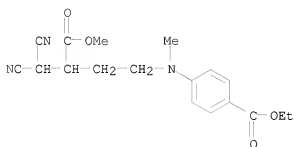
IT 133719-38-7P 133719-41-2P 133719-45-6P  
 133719-47-8P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (preparation and reaction of, in preparation of antitumor agents)

RN 133719-38-7 CAPLUS

CN Benzoic acid, 4-[[4,4-dicyano-3-(methoxycarbonyl)butyl]methylamino]-,

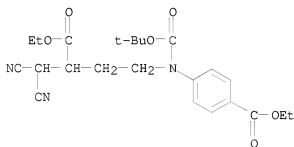
10/923,271

ethyl ester (CA INDEX NAME)



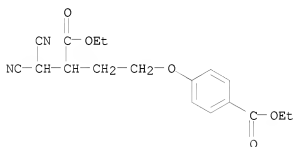
RN 133719-41-2 CAPLUS

CN Benzoic acid, 4-[[4,4-dicyano-3-(ethoxycarbonyl)butyl][(1,1-dimethylethoxy)carbonyl]amino]-, ethyl ester (CA INDEX NAME)



RN 133719-45-6 CAPLUS

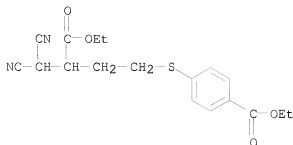
CN Benzoic acid, 4-[[4,4-dicyano-3-(ethoxycarbonyl)butoxy]-, ethyl ester (CA INDEX NAME)



RN 133719-47-8 CAPLUS

CN Benzoic acid, 4-[[4,4-dicyano-3-(ethoxycarbonyl)butyl]thio]-, ethyl ester (CA INDEX NAME)





L4 ANSWER 40 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1991:429194 CAPLUS

DOCUMENT NUMBER: 115:29194

ORIGINAL REFERENCE NO.: 115:5133a,5136a

TITLE: Synthesis of esters of 3,3-dicyano-2-(trifluoromethyl)acrylic acid and their reactions with aryl amines

AUTHOR(S): Tyutin, V. Y.; Chkanikov, N. D.; Kolomiets, A. F.; Fokin, A. V.

CORPORATE SOURCE: Inst. Organoelem. Compd., Moscow, 117813, USSR

SOURCE: Journal of Fluorine Chemistry (1991), 51(3), 323-34

CODEN: JFLCAR; ISSN: 0022-1139

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 115:29194

GI

\* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT \*

AB Title acrylates (NC)2C:C(CF3)CO2R (I; R = Me, Et) were prepared by the condensation of CH2(CN)2 with CF3COCO2R in presence of ZnCl2. Reaction of I with aromatic amines was investigated. Thus, 2,6-dimethylaniline reacted with I in CHCl3 to give adduct II. 2,5-Dimethoxyaniline, and Ph2NH gave similar adducts. o- And m-C6H4(NH2)2 reacted with I to give cyclocondensation products, quinoxalinone III and indoline IV resp.

4-R1C6H4NHNH2 (R1 = H, NO2) gave pyrazolines V on cyclocondensation with I. Reaction of I with 3-aminopyrazole gave pyrazolopyridines VI.

IT 134641-38-6P 134641-39-7P 134641-40-0P

134641-41-1P 134641-42-2P 134641-43-3P

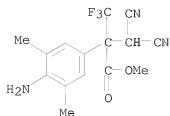
134641-44-4P

RL: SPN (Synthetic preparation); PREP (Preparation) (preparation of)

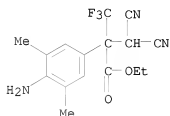
RN 134641-38-6 CAPLUS

CN Benzeneacetic acid, 4-amino- $\alpha$ -(dicyanomethyl)-3,5-dimethyl- $\alpha$ -(trifluoromethyl)-, methyl ester (CA INDEX NAME)

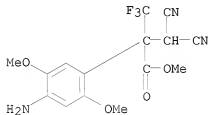
10/923,271



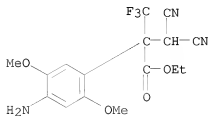
RN 134641-39-7 CAPLUS  
CN Benzeneacetic acid, 4-amino-α-(dicyanomethyl)-3,5-dimethyl-α-(trifluoromethyl)-, ethyl ester (CA INDEX NAME)



RN 134641-40-0 CAPLUS  
CN Benzeneacetic acid, 4-amino-α-(dicyanomethyl)-2,5-dimethoxy-α-(trifluoromethyl)-, methyl ester (CA INDEX NAME)

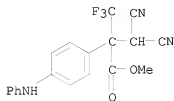


RN 134641-41-1 CAPLUS  
CN Benzeneacetic acid, 4-amino-α-(dicyanomethyl)-2,5-dimethoxy-α-(trifluoromethyl)-, ethyl ester (CA INDEX NAME)



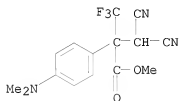
RN 134641-42-2 CAPLUS

CN Benzeneacetic acid,  $\alpha$ -(dicyanomethyl)-4-(phenylamino)- $\alpha$ -(trifluoromethyl)-, methyl ester (CA INDEX NAME)



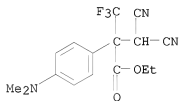
RN 134641-43-3 CAPLUS

CN Benzeneacetic acid,  $\alpha$ -(dicyanomethyl)-4-(dimethylamino)- $\alpha$ -(trifluoromethyl)-, methyl ester (CA INDEX NAME)



RN 134641-44-4 CAPLUS

CN Benzeneacetic acid,  $\alpha$ -(dicyanomethyl)-4-(dimethylamino)- $\alpha$ -(trifluoromethyl)-, ethyl ester (CA INDEX NAME)



L4 ANSWER 41 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1991:247080 CAPLUS

DOCUMENT NUMBER: 114:247080

ORIGINAL REFERENCE NO.: 114:41709a,41712a

TITLE: Reaction of  $\alpha,\beta$ -unsaturated nitriles with phosphorus ylides

AUTHOR(S): Abdou, Wafaa M.; Ganoub, Neven A. F.

CORPORATE SOURCE: Natl. Res. Cent., Dokki, Egypt

SOURCE: Chemistry & Industry (London, United Kingdom) (1991), (6), 217-18

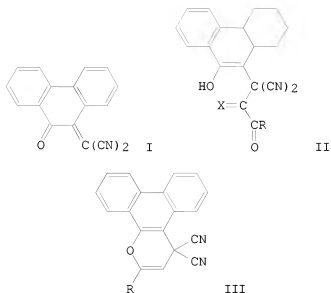
CODEN: CHINAG; ISSN: 0009-3068

DOCUMENT TYPE: Journal

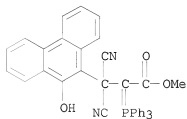
LANGUAGE: English

OTHER SOURCE(S): CASREACT 114:247080

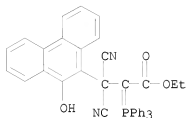
GI



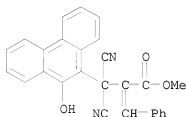
- AB Reaction of unsatd. nitrile I with ROCC-HP+Ph<sub>3</sub> (R = OMe, OEt, Ph) in benzene at 25° gave 75-80% addition products II (X = PPh<sub>3</sub>). On heating II (X = PPh<sub>3</sub>) to 200° they underwent an intramol. Wittig reaction to give arenopyrans III. Heating phosphorane II (R = OMe, X = PPh<sub>3</sub>) with BzH gave II (X = CHPh).
- IT 133973-19-0P 133973-20-3P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (preparation and intramol. Wittig reaction of)
- RN 133973-19-0 CAPLUS
- CN 9-Phenanthrenepranoic acid,  $\beta,\beta$ -dicyano-10-hydroxy- $\alpha$ -(triphenylphosphoranylidene)-, methyl ester (CA INDEX NAME)



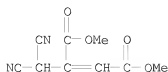
- RN 133973-20-3 CAPLUS
- CN 9-Phenanthrenepranoic acid,  $\beta,\beta$ -dicyano-10-hydroxy- $\alpha$ -(triphenylphosphoranylidene)-, ethyl ester (CA INDEX NAME)



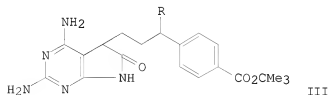
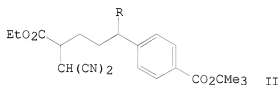
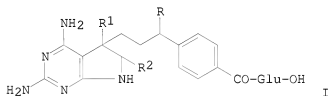
IT 133973-25-8P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)  
 RN 133973-25-8 CAPLUS  
 CN 9-Phenanthrene-10-carboxylic acid,  $\beta,\beta$ -dicyano-10-hydroxy- $\alpha$ -  
 (phenylmethylene)-, methyl ester (CA INDEX NAME)



L4 ANSWER 42 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1991:121991 CAPLUS  
 DOCUMENT NUMBER: 114:121991  
 ORIGINAL REFERENCE NO.: 114:20773a,20776a  
 TITLE: Reactions of malononitrile with acetylenic esters and  
 ketones [Erratum to document cited in  
 CA113(25):231170y]  
 AUTHOR(S): Kandeel, Kamal A.; Vernon, John M.; Dransfield, Trevor  
 A.; Fouli, Fouli A.; Youssef, Ahmed S. A.  
 CORPORATE SOURCE: Dep. Chem., Univ. York, Heslington, York, YO1 5DD, UK  
 SOURCE: Journal of Chemical Research, Synopses (1990  
 ), (12), 406  
 CODEN: JRPSDC; ISSN: 0308-2342  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 AB An error in the structure for compound 13 has been corrected The error was not  
 reflected in the abstract or the index entries.  
 IT 130747-61-4P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of (Erratum))  
 RN 130747-61-4 CAPLUS  
 CN 2-Butenedioic acid, 2-(dicyanomethyl)-, dimethyl ester (9CI) (CA INDEX  
 NAME)



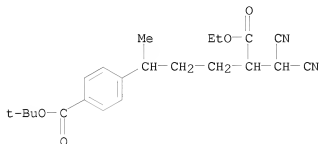
L4 ANSWER 43 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1991:82446 CAPLUS  
 DOCUMENT NUMBER: 114:82446  
 ORIGINAL REFERENCE NO.: 114:14101a,14104a  
 TITLE: Novel pyrrolo[2,3-d]pyrimidine antifolates: synthesis and antitumor activities  
 AUTHOR(S): Miwa, Tetsuo; Hitaka, Takenori; Akimoto, Hiroshi; Nomura, Hiroaki  
 CORPORATE SOURCE: Res. Dev. Div., Takeda Chem. Ind., Ltd., Osaka, 532, Japan  
 SOURCE: Journal of Medicinal Chemistry (1991), 34(2), 555-60  
 CODEN: JMCMAR; ISSN: 0022-2623  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 114:82446  
 GI



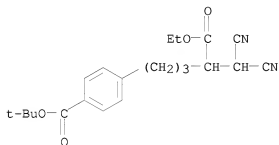
AB Title compds. I (R = H, Me; R1 = H, Et; R2R3 = bond, R2 = R3 = H) were prepared as antifolates. A key step was the cyclocondensation of dicyano compound II (R = H, Me) with guanidine-HCl to give pyrrolo[2,3-d]pyrimidines

III. III were prepared in several steps from p-RCOC6H4CO2CMe2 and CH3CH:CHCO2Et or BrCH2CH:CHCO2Et. These antifolates were more growth-inhibitory by about 1 order of magnitude than methotrexate (MTX) against KB human epidermoid carcinoma cells and A549 human nonsmall cell lung carcinoma cells in in vitro culture.

IT 125991-47-1P 130351-33-6P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (preparation and cyclocondensation of, with guanidine)  
 RN 125991-47-1 CAPLUS  
 CN Benzenepentanoic acid,  $\alpha$ -(dicyanomethyl)-4-[(1,1-dimethylethoxy)carbonyl]-8-methyl-, ethyl ester (CA INDEX NAME)

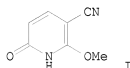


RN 130351-33-6 CAPLUS  
 CN Benzenepentanoic acid,  $\alpha$ -(dicyanomethyl)-4-[(1,1-dimethylethoxy)carbonyl]-, ethyl ester (CA INDEX NAME)



L4 ANSWER 44 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1990:631170 CAPLUS  
 DOCUMENT NUMBER: 113:231170  
 ORIGINAL REFERENCE NO.: 113:39001a,39004a  
 TITLE: Reactions of malononitrile with acetylenic esters and ketones  
 AUTHOR(S): Kandeel, Kamal A.; Vernon, John M.; Dransfield, Trevor A.; Fouli, Fouli A.; Youssef, Ahmed S. A.  
 CORPORATE SOURCE: Dep. Chem., Univ. York, Heslington/York, YO1 5DD, UK  
 SOURCE: Journal of Chemical Research, Synopses (1990), (9), 276-7  
 CODEN: JRPSDC; ISSN: 0308-2342

DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 113:231170  
 GI



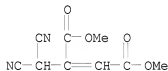
AB The addition of malononitrile to acetylenic esters and acetylenic ketones catalyzed by sodium alkoxides gave 3- and 5-cyano-2-pyridones, e.g., I, 2-cyano- and 2,6-dicyanoaniline, and other products.

IT 130747-61-4P

RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)

RN 130747-61-4 CAPLUS

CN 2-Butenedioic acid, 2-(dicyanomethyl)-, dimethyl ester (9CI) (CA INDEX NAME)



L4 ANSWER 45 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1990:497432 CAPLUS

DOCUMENT NUMBER: 113:97432

ORIGINAL REFERENCE NO.: 113:16453a,16456a

TITLE: Quinolone antibacterial agents substituted at the 7-position with spiroamines. Synthesis and structure-activity relationships

AUTHOR(S): Culbertson, Townley P.; Sanchez, Joseph P.; Gambino, Laura; Sesnie, Josephine A.

CORPORATE SOURCE: Parke-Davis Pharm. Res. Div., Warner-Lambert Co., Ann Arbor, MI, 48105, USA

SOURCE: Journal of Medicinal Chemistry (1990), 33(8), 2270-5

CODEN: JMCMAR; ISSN: 0022-2623

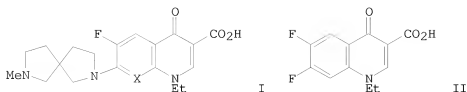
DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 113:97432

GI





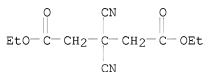
AB Fluoroquinolone antibacterials having the 7-position (10-position of pyridobenzoxazines) substituted with 2,7-diazaspiro[4.4]nonane, 1,7-diazaspiro[4.4]nonane, or 2,8-diazaspiro[5.5]undecane (e.g. I (X = CF, CH, N) were prepared and their biol. activities were compared with piperazine and pyrrolidine substituted analogs. Most exhibited potent Gram-pos. and Gram-neg. activity, especially when side chain was N-alkylated. Thus, the quinolinecarboxylic acid II was treated with 2-methyl-2,7-diazaspiro[4.4]nonane to give I (X = CH).

IT 77415-69-1

RL: RCT (Reactant); RACT (Reactant or reagent)  
(reductive cyclization of)

RN 77415-69-1 CAPLUS

CN Pentanedioic acid, 3,3-dicyano-, diethyl ester (9CI) (CA INDEX NAME)



L4 ANSWER 46 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1990:235871 CAPLUS

DOCUMENT NUMBER: 112:235871

ORIGINAL REFERENCE NO.: 112:39805a,39806a

TITLE: New gem-dicyanocyclobutane-containing hydroxyesters

AUTHOR(S): Mori, Shoji; Kakuchi, Toyoji; Padias, Anne Buyle;

Hall, H. K., Jr.

CORPORATE SOURCE: Chem. Dep., Univ. Arizona, Tucson, AZ, 85721, USA

SOURCE: Journal of Polymer Science, Part A: Polymer Chemistry

(1990), 28(3), 551-8

CODEN: JPACEC; ISSN: 0887-624X

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Six gem-dicyanocyclobutanes containing carbomethoxy and hydroxyl/acetoxyl functions were synthesized by cycloaddn. of the appropriate vinyl ethers or alkoxystyrenes to Me  $\beta,\beta$ -dicyanoacrylate. They were too thermally liable to allow polycondensation to potentially piezoelec. linear polyesters.

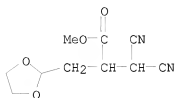
IT 127396-28-5P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation and attempted polymerization of)

RN 127396-28-5 CAPLUS

CN 1,3-Dioxolane-2-propanoic acid,  $\alpha$ -(dicyanomethyl)-, methyl ester

(CA INDEX NAME)



L4 ANSWER 47 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1990:158968 CAPLUS

DOCUMENT NUMBER: 112:158968

ORIGINAL REFERENCE NO.: 112:26887a,26890a

TITLE: Preparation of N-[(pyrrolopyrimidinylalkyl)benzoyl]glutamates as neoplasm inhibitors

INVENTOR(S): Akimoto, Hiroshi; Hitaka, Takenori; Miwa, Tetsuo

PATENT ASSIGNEE(S): Takeda Chemical Industries, Ltd., Japan

SOURCE: Eur. Pat. Appl., 24 pp.

CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 334636	A2	19890927	EP 1989-302851	19890322 <--
EP 334636	A3	19910502		
EP 334636	B1	19961023		
R: AT, BE, CH, DE, ES, FR, GB, GR, IT, LI, LU, NL, SE				
NO 8901206	A	19890925	NO 1989-1206	19890320 <--
NO 169490	B	19920323		
NO 169490	C	19920701		
US 4997838	A	19910305	US 1989-326901	19890321 <--
DK 8901437	A	19890925	DK 1989-1437	19890322 <--
DK 173980	B1	20020325		
AT 144513	T	19961115	AT 1989-302851	19890322 <--
ES 2092994	T3	19961216	ES 1989-302851	19890322 <--
CA 1340794	C	19991019	CA 1989-594699	19890323 <--
CN 1037513	A	19891129	CN 1989-101681	19890324 <--
CN 1029970	B	19951011		
HU 51624	A2	19900528	HU 1989-1517	19890324 <--
HU 203105	B	19910528		
JP 02167281	A	19900627	JP 1989-72235	19890324 <--
JP 07005599	B	19950125		
HU 55396	A2	19910528	HU 1990-8458	19890324 <--
HU 215928	B	19990329		
US 5106974	A	19920421	US 1990-578258	19900906 <--
NO 9100661	A	19890925	NO 1991-661	19910219 <--
NO 178304	B	19951120		
NO 178304	C	19960228		
US 5296600	A	19940322	US 1992-824106	19920122 <--

US 5539113  
PRIORITY APPLN. INFO.:

A 19960723

US 1993-161533

19931206 <--

JP 1988-71149

A 19880324

JP 1988-245379

A 19880929

NO 1989-1206

A1 19890320

US 1989-326901

A3 19890321

US 1990-578258

A3 19900906

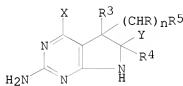
US 1992-824106

A3 19920122

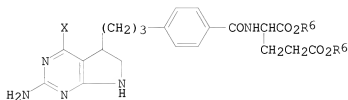
OTHER SOURCE(S):

CASREACT 112:158968; MARPAT 112:158968

GI



I



III

AB The title compds. [I; R = H, F, alkyl, alkenyl, alkynyl; R3, R4 = H; R3R4 = bond; R5 = C6H4(CONHCHR1CH2CH2R2)-4; R1,R2 = (un)esterified CO2H; X = NH2, OH; Y = H, NH2, OH; n = 2-4] were prepared. Thus, 4-(Me3CO2C)C6H4(CH2)3CH[CH(CN)2]CO2Me (preparation given) was refluxed 28 h with (H2N)2C:NH.HCl in Me3COH containing Me3COK to give I [R = R3 = H, R5 = C6H4(CO2CMe3)-4, X = NH2, n = 3] (II; R4Y = O) which was hydrogenated to II (R4 = Y = H). The latter was hydrolyzed and the product condensed with di-Et L-glutamate to give title compound III (R6 = Et, X = NH2) which was hydrolyzed to III (R6 = H, X = OH) which had IC50 of 0.0006 µg/mL against human nasopharyngeal cancer KB cells in vitro.

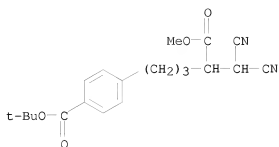
IT 125991-38-0P 125991-47-1P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

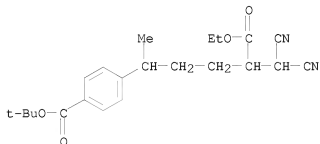
(preparation and reaction of, in preparation of neoplasm inhibitors)

RN 125991-38-0 CAPLUS

CN Benzenepentanoic acid, α-(dicyanomethyl)-4-[(1,1-dimethylethoxy)carbonyl]-, methyl ester (CA INDEX NAME)



RN 125991-47-1 CAPLUS  
 CN Benzenepentanoic acid,  $\alpha$ -(dicyanomethyl)-4-[(1,1-dimethylethoxy)carbonyl]-8-methyl-, ethyl ester (CA INDEX NAME)



L4 ANSWER 48 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1989:415389 CAPLUS  
 DOCUMENT NUMBER: 111:15389  
 ORIGINAL REFERENCE NO.: 111:2625a,2628a  
 TITLE: Color photothermographic elements containing leuco compounds  
 INVENTOR(S): Sakizadeh, Kumars; Weigel, David C.; Grieve, Duncan; Poon, Stephen S. C.; Thien, Tran V.  
 PATENT ASSIGNEE(S): Minnesota Mining and Manufacturing Co., USA  
 SOURCE: Eur. Pat. Appl., 35 pp.  
 CODEN: EPXXDW  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 294099	A2	19881207	EP 1988-304771	19880526 <--
EP 294099	A3	19890531		
EP 294099	B1	19930818		
R: BE, DE, FR, GB, IT				
US 4883747	A	19891128	US 1988-200665	19880531 <--
CA 1331107	C	19940802	CA 1988-568396	19880602 <--
AU 8817345	A	19881208	AU 1988-17345	19880603 <--
AU 606162	B2	19910131		

JP 02032332 A 19900202 JP 1988-145566 19880613 <--  
 JP 2590204 B2 19970312  
 US 4923792 A 19900508 US 1989-368566 19890620 <--  
 PRIORITY APPLN. INFO.: GB 1987-12961 A 19870603  
 US 1988-200665 A1 19880531

OTHER SOURCE(S): CASREACT 111:15389; MARPAT 111:15389

AB A photothermog. material comprises Ag halide in reactive association with a Ag salt of an organic acid and a color-generating reducing agent which is a leuco compound oxidizable by Ag ions into a colored dye of the formula  $\text{ArR1C}:(\text{C}(\text{R5})\text{C}(\text{R4}))\text{nCR2R3}$  [n = 0-2; R1 = H, CN, C1-5 alkyl, aryl, CO2R6; R6 = C1-5 alkyl or aryl; R2, R3 = CN, NO2, CO2R6, SO2R6, COR6; R3 and R2 may combine together to form a ring; R4, R5 = H, CN, C1-5 alkyl, or R4 and R5 together may form a ring; Ar = thienyl, furyl, phenyl]. The material produces images with improved color stability. Thus, a green-yellow image was produced with a photothermog. material incorporating leuco form of (p-dimethylaminobenzylidene)dimethylbarbituric acid.

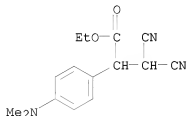
IT 121246-61-5

RL: USES (Uses)

(photothermog. material containing, for improved image stability)

RN 121246-61-5 CAPLUS

CN Benzeneacetic acid,  $\alpha$ -(dicyanomethyl)-4-(dimethylamino)-, ethyl ester (CA INDEX NAME)



L4 ANSWER 49 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1988:570272 CAPLUS

DOCUMENT NUMBER: 109:170272

ORIGINAL REFERENCE NO.: 109:28239a,28242a

TITLE: Synthesis and cognition-activating properties of some mono- and bicyclic lactam derivatives

AUTHOR(S): Altomare, Cosimo; Carotti, Angelo; Casini, Giovanni; Cellamare, Saverio; Ferappi, Marcello; Gavuzzo, Enrico; Mazza, Fernando; Pantaleoni, Giancarlo; Giorgi, Raffaele

CORPORATE SOURCE: Dip. Farm.-Chim., Univ. Bari, Bari, Italy

SOURCE: Journal of Medicinal Chemistry (1988),

31(11), 2153-8

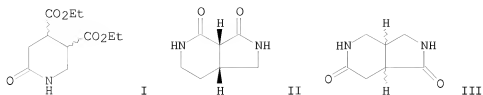
CODEN: JMCMAR; ISSN: 0022-2623

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 109:170272

GI



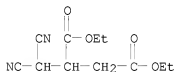
AB Upon reductive cyclization cyano esters  $\text{EtO}_2\text{CCH}_2\text{CH}(\text{CO}_2\text{Et})\text{CHRCN}$  ( $\text{R} = \text{CO}_2\text{Et}$ , cyano) and  $\text{NCCH}_2\text{CH}(\text{CN})\text{CH}(\text{CO}_2\text{Et})_2$  yielded piperidones and perhydropyrrolo[3,4-c]pyridine lactams I, II and III, resp. generally as a mixture of diastereomeric cis-trans forms. X-ray crystallog. anal. were carried out on cis-II and III. A series of neuropsychopharmacol. tests performed on I, II, and III indicated that they are generally nontoxic even at high doses (up to 1000 mg/kg i.p.)<sup>9</sup>. The cognition activating properties of lactams cis- and trans-I, cis-II, and III were evaluated in enhancing retention for passive avoidance learning in rats without and after electroconvulsive shock (ECS); compds. cis-I and III were found to be more potent than piracetam in the amnesia-reversal testing.

IT 82584-86-9

RL: RCT (Reactant); RACT (Reactant or reagent)  
(reductive cyclization of, cyclic lactams from)

RN 82584-86-9 CAPLUS

CN Butanedioic acid, (dicyanomethyl)-, diethyl ester (9CI) (CA INDEX NAME)



L4 ANSWER 50 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1988:94172 CAPLUS

DOCUMENT NUMBER: 108:94172

ORIGINAL REFERENCE NO.: 108:15475a,15478a

TITLE: Addition of ylidenemalononitriles onto dimethyl acetylenedicarboxylate

AUTHOR(S): Gewald, Karl; Hain, Ute; Gruner, Margit

CORPORATE SOURCE: Sekt. Chem., Tech. Univ. Dresden, Dresden, DDR-8027, Ger. Dem. Rep.

SOURCE: Zeitschrift fuer Chemie (1987), 27(1), 32-4

CODEN: ZECEAL; ISSN: 0044-2402

Journal

DOCUMENT TYPE:

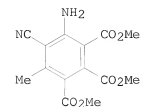
German

LANGUAGE:

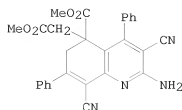
OTHER SOURCE(S):

CASREACT 108:94172

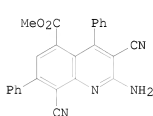
GI



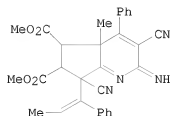
II



III



IV



V

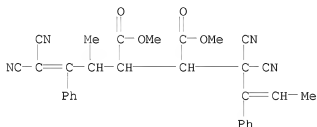
AB (NC)2C:CMech2CO2Me and MeO2CC.tplbond.CCO2Me (I) in the presence of K<sub>2</sub>CO<sub>3</sub> cycloadded to give 49% aniline II. (NC)2C:CPhMe and I, treated with Et<sub>3</sub>N, gave 30% dihydroquinoline III, which was aromatized by heating at 270° in Na-MeOH to 80% quinolinecarboxylate IV. (NC)2C:CPhEt and I gave 29% (NC)2C:CPhCHMeCH(CO<sub>2</sub>Me)CH(CO<sub>2</sub>Me)C(CN)2CPh:CHMe, which cyclized to cyclopentapyridinedicarboxylate V.

IT 112754-03-7P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(preparation and thermal intramol. cyclization of)

RN 112754-03-7 CAPLUS

CN Butanedioic acid, 2-(3,3-dicyano-1-methyl-2-phenyl-2-propenyl)-3-(1,1-dicyano-2-phenyl-2-butenyl)-, dimethyl ester (9CI) (CA INDEX NAME)



L4 ANSWER 51 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1987:597249 CAPLUS

DOCUMENT NUMBER: 107:197249

ORIGINAL REFERENCE NO.: 107:31627a,31630a

TITLE: Influence of the solvent on the nature of a

tetramethylene biradical intermediate

Padias, Anne Buyle; Hall, H. K., Jr.

AUTHOR(S): Chem. Dep., Univ. Arizona, Tucson, AZ, 85721, USA

SOURCE: Journal of Organic Chemistry (1987), 52(20),

4536-9

CODEN: JOCEAH; ISSN: 0022-3263

DOCUMENT TYPE:

Journal

LANGUAGE:

English

OTHER SOURCE(S):

CASREACT 107:197249

AB In the spontaneous thermal reactions of p-methoxystyrene and Me 3,3-dicyanoacrylate, several reaction products are observed: a 1/1 alternating copolymer, a double Diels-Alder adduct, and the cyclobutane adduct. In dipolar aprotic solvents, no polymerization occurs, and the double Diels-Alder adduct is favored; in protic polar solvents cyclobutane formation competes with copolymer. In nonpolar solvents, copolymer dominates. A biradical tetramethylene species is proposed as the key intermediate. In polar solvents, this biradical exhibits considerable polar character, and Coulombic attraction between the termini favors the coiled or gauche conformation, leading preferentially to cycloadducts. In nonpolar solvents, the trans conformation initiates the polymerization. The

main factors influencing the products are the solvent polarity and the ability of the solvent to interact with the biradical.

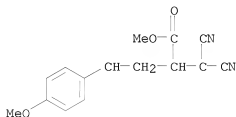
IT 110193-00-5

RL: PRP (Properties)

(conformation and spin and electron d. of, solvent effects on)

RN 110193-00-5 CAPLUS

CN 1,4-Butanediyl, 1,1-dicyano-2-(methoxycarbonyl)-4-(4-methoxyphenyl)- (9CI)  
(CA INDEX NAME)



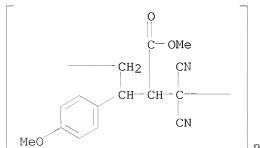
IT 110193-05-0P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)

RN 110193-05-0 CAPLUS

CN Poly[1,1-dicyano-2-(methoxycarbonyl)-3-(4-methoxyphenyl)-1,4-butanediyl]  
(9CI) (CA INDEX NAME)





L4 ANSWER 52 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1987:196834 CAPLUS

DOCUMENT NUMBER: 106:196834

ORIGINAL REFERENCE NO.: 106:31929a,31932a

TITLE: Cationic polymerization of nitrogen-containing electron-rich vinyl monomers by electrophilic olefins and their cyclobutane cycloadducts

AUTHOR(S): Abdelkader, Mohamed; Padias, Anne Buyle; Hall, H. K., Jr.

CORPORATE SOURCE: Chem. Dep., Univ. Arizona, Tucson, AZ, 85721, USA

SOURCE: Macromolecules (1987), 20(5), 944-8

CODEN: MAMOBX; ISSN: 0024-9297

DOCUMENT TYPE: Journal

LANGUAGE: English

AB The major pathways for the reactions of very electron-rich N-containing olefins with several electrophilic olefins were studied. N-Ethyl-3-vinylcarbazole (I) [1486-07-3], N-vinylcarbazole (II) [1484-13-5], and p-(dimethylamino)styrene (III) [2039-80-7] underwent kinetic cyclobutane formation with an electrophilic olefin without a leaving group, Me  $\beta$ , $\beta$ -dicyanoacrylate (IV) [82849-50-1], and one with a weak  $\beta$ -leaving group, tetracyanoethylene (V) [670-54-2]. The third electrophilic olefin,  $\beta$ , $\beta$ -dicyanovinyl chloride (VI) [10472-09-0], had a strong  $\beta$ -leaving group and readily initiated the cationic polymerization of I and II and oligomerization of III. If an excess

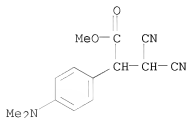
of donor olefin was used, IV, V, and VI all initiated cationic homopolymn. of I and II, while III only led to oligomers, as it did with conventional Broensted initiators. Cationic initiation by their own cyclobutane adducts was observed for the very electron-rich monomers I and II. Postcyanovinylation of the formed polymers by the electrophilic olefins occurred. Incorporation of a  $\beta$ -leaving group enhanced the initiating ability of the electrophilic olefins and N-carbazyl and N-ethyl-3-carbazyl were overall the most effective donor substituents favoring cationic homopolymn.

IT 107540-79-4P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of, from dimethylaniline and dicyanovinyl compound)

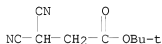
RN 107540-79-4 CAPLUS

CN Benzeneacetic acid,  $\alpha$ -(dicyanomethyl)-4-(dimethylamino)-, methyl ester (CA INDEX NAME)

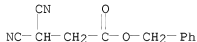
L4 ANSWER 53 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1987:33439 CAPLUS

DOCUMENT NUMBER: 106:33439  
 ORIGINAL REFERENCE NO.: 106:5623a,5626a  
 TITLE: Synthesis of novel symmetric diamino acids  
 AUTHOR(S): Reddy, P. Anantha; Erickson, Bruce W.  
 CORPORATE SOURCE: Rockefeller Univ., New York, NY, 10021, USA  
 SOURCE: Pept.: Struct. Funct., Proc. Am. Pept. Symp., 9th (1985), 453-6  
 CODEN: 54ZNAJ  
 DOCUMENT TYPE: Conference  
 LANGUAGE: English  
 AB Sym diamino acids (H2NCH2)2CHCH2CO2H (Aab) 3,5-(H2NCH2X)2C6H3CO2H [X = null, CH2 (Bab)] were prepared from (NC)2CHCH2CO2CMe3 and 3,5-(BrCH2)2C6H3CO2Me. The N,N-bis(tert-butoxycarbonyl) derivative of Aab couples efficiently during solid-phase peptide synthesis. The corresponding derivative of Bab is used in the synthesis of the protein betabellin.  
 IT 105995-37-7P 105995-39-9P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (preparation and hydrogenation of)  
 RN 105995-37-7 CAPLUS  
 CN Propanoic acid, 3,3-dicyano-, 1,1-dimethylethyl ester (CA INDEX NAME)



RN 105995-39-9 CAPLUS  
 CN Propanoic acid, 3,3-dicyano-, phenylmethyl ester (CA INDEX NAME)



L4 ANSWER 54 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1986:496793 CAPLUS  
 DOCUMENT NUMBER: 105:96793  
 ORIGINAL REFERENCE NO.: 105:15633a,15636a  
 TITLE: Zwitterionic tetramethylenes as the common intermediates in the cycloaddition and polymerization reactions of N-vinylcarbazole with electrophilic tetrasubstituted ethylenes: a new explanation for charge-transfer initiation  
 AUTHOR(S): Gotoh, Tetsuya; Padias, Anne Buyle; Hall, H. K., Jr.  
 CORPORATE SOURCE: Chem. Dep., Univ. Arizona, Tucson, AZ, 85721, USA  
 SOURCE: Journal of the American Chemical Society (1986), 108(16), 4920-31  
 CODEN: JACSAT; ISSN: 0002-7863  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 105:96793

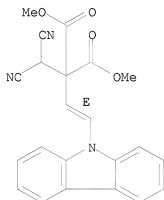
AB The reactions of N-vinylcarbazole (I) with electrophilic tetrasubstituted ethylenes were examples of reactions whose outcomes are manipulated by changes in concentration, structure, and working procedure to form either small mols. (cyclobutanes, 1-butenes) or poly(vinylcarbazole). Equivalent concns. and evaporative workup (organic chemists' conditions) lead to small mols.; a large excess of I and precipitative workup give polymer. The mechanism involves gauche and trans zwitterionic tetramethylenes as intermediates. The former gives cyclobutane reversibly. The latter gives 1-butenes intramol. or adds monomers to form cyclohexanes or eventually polymer. The organic chemical and polymer chemical are unified on this basis. Extensive stereochem. and kinetic support for these propositions is given. Two other proposed mechanisms for these charge-transfer initiations are excluded.

IT 96735-90-9P 102852-12-0P 102852-13-1P  
102852-14-2P 102852-38-0P 102852-39-1P  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 96735-90-9 CAPLUS

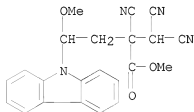
CN Propanedioic acid, [2-(9H-carbazol-9-yl)ethenyl](dicyanomethyl)-, dimethyl ester, (E)- (9CI) (CA INDEX NAME)

Double bond geometry as shown.



RN 102852-12-0 CAPLUS

CN 9H-Carbazole-9-butanoic acid,  $\alpha$ -cyano- $\alpha$ -(dicyanomethyl)- $\gamma$ -methoxy-, methyl ester (CA INDEX NAME)

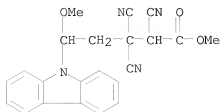


RN 102852-13-1 CAPLUS

CN 9H-Carbazole-9-pentanoic acid,  $\alpha,\beta$ -tricyano- $\delta$ -

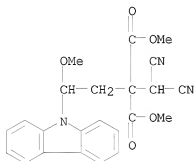
10/923,271

methoxy-, methyl ester (CA INDEX NAME)



RN 102852-14-2 CAPLUS

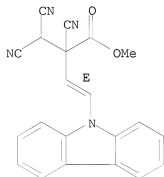
CN Propanedioic acid, [2-(9H-carbazol-9-yl)-2-methoxyethyl] (dicyanomethyl)-, dimethyl ester (9CI) (CA INDEX NAME)



RN 102852-38-0 CAPLUS

CN 3-Butenoic acid, 4-(9H-carbazol-9-yl)-2-cyano-2-(dicyanomethyl)-, methyl ester, (E)- (9CI) (CA INDEX NAME)

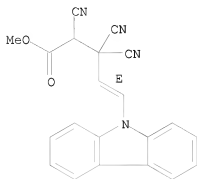
Double bond geometry as shown.



RN 102852-39-1 CAPLUS

CN 4-Pentenoic acid, 5-(9H-carbazol-9-yl)-2,3,3-tricyano-, methyl ester, (E)- (9CI) (CA INDEX NAME)

Double bond geometry as shown.



L4 ANSWER 55 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1985:406747 CAPLUS

DOCUMENT NUMBER: 103:6747

ORIGINAL REFERENCE NO.: 103:1225a,1228a

TITLE: Zwitterionic tetramethylene intermediates: a new interpretation for "charge-transfer" initiation  
Hall, H. K., Jr.; Gotoh, T.

AUTHOR(S): Dep. Chem., Univ. Arizona, Tucson, AZ, 85721, USA

CORPORATE SOURCE: Polymer Preprints (American Chemical Society, Division of Polymer Chemistry) (1985), 26(1), 34-5

SOURCE: CODEN: ACPPAY; ISSN: 0032-3934

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Investigation of the initiation mechanism in polymerization of N-vinylcarbazole (I) [1484-13-5] in the presence of tetracyanoethylene [670-54-2] or di-Me 2,2-dicyanoethylene-1,1-dicarboxylate [82849-49-8] showed that neither the I-cyano compound charge transfer complexes nor the ion-radical pairs formed from them initiated polymerization. The initiating species was the gauche or trans tetramethylene zwitterion formed as an intermediate from the charge-transfer complex. This finding indicated that cyclobutanes initiated vinyl polymerization. The mechanism and the kinetics of the zwitterionic initiation were discussed.

IT 96735-90-9

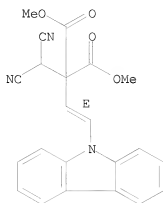
RL: CAT (Catalyst use); USES (Uses)

(catalysts, for vinylcarbazole polymerization)

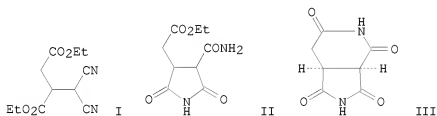
RN 96735-90-9 CAPLUS

CN Propanedioic acid, [2-(9H-carbazol-9-yl)ethenyl](dicyanomethyl)-, dimethyl ester, (E)- (9CI) (CA INDEX NAME)

Double bond geometry as shown.



L4 ANSWER 56 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1983:488082 CAPLUS  
 DOCUMENT NUMBER: 99:88082  
 ORIGINAL REFERENCE NO.: 99:13589a,13592a  
 TITLE: Tetraoxo derivatives of perhydropyrrolo[3,4-c]pyridine  
 AUTHOR(S): Ferappi, M.; Carotti, A.; Casini, G.; De Laurentis, N.; Giardina, D.; Cingolani, G. M.; Gavuzzo, E.; Mazza, F.  
 CORPORATE SOURCE: Ist. Chim. Farm. Tossicol., Univ. Bari, Bari, 70126, Italy  
 SOURCE: Journal of Heterocyclic Chemistry (1983), 20(2), 439-46  
 CODEN: JHTCAD; ISSN: 0022-152X  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 99:88082  
 GI



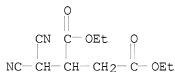
AB Michael adducts from di-Et furmarate with malonic esters or nitriles were cyclized to succinimide intermediates which, after glutarimide ring closure, afforded several N-Me and N-benzyl derivs. of cis-1,3,4,6-tetraoxoperhydropyrrolo[3,4-c]pyridine whose configuration was demonstrated by x-ray crystal structure anal. Thus, treating the adduct I with H2SO4 gave succinimide II which was treated with NaOEt in EtOH or tosyl acid in xylene to give pyrrolopyridine III.  
 IT 82584-86-9P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT

(Reactant or reagent)

(preparation and cyclization of, pyrrolidine from)

RN 82584-86-9 CAPLUS

CN Butanedioic acid, (dicyanomethyl)-, diethyl ester (9CI) (CA INDEX NAME)



L4 ANSWER 57 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1982:582893 CAPLUS

DOCUMENT NUMBER: 97:182893

ORIGINAL REFERENCE NO.: 97:30617a,30620a

TITLE: Dimethyl 1,1-dicyanoethene-2,2-dicarboxylate, a new electrophilic olefin

AUTHOR(S): Hall, H. K., Jr.; Sentman, R. C.

CORPORATE SOURCE: Dep. Chem., Univ. Arizona, Tucson, AZ, 85721, USA

SOURCE: Journal of Organic Chemistry (1982), 47(23), 4572-7

CODEN: JOCEAH; ISSN: 0022-3263

DOCUMENT TYPE: Journal

LANGUAGE: English

AB dimethyl 1,1-dicyanoethene-2,2-dicarboxylate (I) [82849-49-8] was synthesized via a Knoevenagel condensation. I spontaneously copolymerizes with electron-rich olefins such as styrene [100-42-5] and p-methylstyrene [622-97-9]. In the copolymn., the bulky growing styryl radicals add to the dicyano-bearing carbon of I. Cyclobutane adducts are obtained in thermal reactions with styrene, p-methylstyrene, p-methoxystyrene [637-69-4], and vinyl ethers via a tetramethylene intermediate. Bond formation occurs at the diester end of I due to the greater stabilization provided by the dicyano group and the minimal steric requirements of the attacking methylene.

IT 82849-58-9P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)  
 (preparation and NMR spectra of)

RN 82849-58-9 CAPLUS

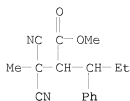
CN Benzenepropanoic acid,  $\alpha$ -(1,1-dicyanoethyl)- $\beta$ -ethyl-, methyl ester, polymer with ethenylbenzene (9CI) (CA INDEX NAME)

CM 1

CRN 82917-40-6

CMF C16 H18 N2 O2

10/923,271



CM 2

CRN 100-42-5

CMF C8 H8



L4 ANSWER 58 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1982:472220 CAPLUS

DOCUMENT NUMBER: 97:72220

ORIGINAL REFERENCE NO.: 97:12085a,12088a

TITLE: Contribution to the synthesis of the glutarimides.

III

AUTHOR(S): Victory, Pedro; Jover, Jose Maria; Sempere, Julian

CORPORATE SOURCE: Dep. Quim. Org., Inst. Quim. Sarria, Barcelona, Spain

SOURCE: Afinidad (1981), 38(376), 491-5

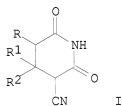
CODEN: AFINAE; ISSN: 0001-9704

DOCUMENT TYPE: Journal

LANGUAGE: Spanish

OTHER SOURCE(S): CASREACT 97:72220

GI



I

AB Glutarimides I [R = R1 = H, R2 = CO2Et, Ph, 3-furyl, 2-thienyl, Me; R =

cyano, R1R2 = (CH2)5; R = Me, R1 = R2 = H] were prepared by treating

CH2(CN)2 with R1R2C:CRCO2Et with or without isolation of

(NC)2CHCR1R2CHRCO2Et, and acid hydrolysis of the enol ethers.

Alternatively R1R2C:CRCO2Et was cyclized with NCCH2CONH2.

IT 82584-86-9P

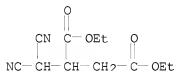
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
(Reactant or reagent)



(preparation and cyclization of)

RN 82584-86-9 CAPLUS

CN Butanedioic acid, (dicyanomethyl)-, diethyl ester (9CI) (CA INDEX NAME)



L4 ANSWER 59 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1981:442180 CAPLUS

DOCUMENT NUMBER: 95:42180

ORIGINAL REFERENCE NO.: 95:7221a,7224a

TITLE: Absolute configuration of 2,7-diazaspiro[4,4]nonane.  
A reassignmentAUTHOR(S): Overberger, C. G.; Wang, David Wei; Hill, Richard K.;  
Krow, Grant R.; Ladner, David W.CORPORATE SOURCE: Macromol. Res. Cent., Univ. Michigan, Ann Arbor, MI,  
48109, USASOURCE: Journal of Organic Chemistry (1981), 46(13),  
2757-64

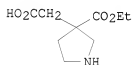
CODEN: JOCEAH; ISSN: 0022-3263

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 95:42180

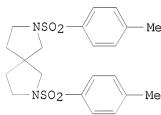
GI



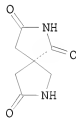
II



III



IV



V

AB The absolute configuration of the axially dissym. spirane 2,7-diazaspiro[4,4]nonane (I), was elucidated as (R)-(-), (S)-(+) in CHCl<sub>3</sub> by synthesis of both enantiomers from the centrodissym. intermediate II; the configuration of (R)-(-)-II was correlated with that of (S)-HO<sub>2</sub>CCHMeEtCH<sub>2</sub>CO<sub>2</sub>H through the substituted pyrrolidine III. The

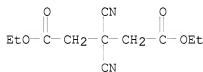
configuration thus established for the sulfonamide derivative IV is opposite to that derived earlier (Krow, G. and Hill, R. K., 1968). The source of the original error lies in the preparation of spiroimide V, which is accompanied by almost total racemization when carried out at high temps. A more direct, efficient synthesis of I is described, followed by resolution with dinitrodiphenic acid to give the optically pure enantiomers. Lowe's rule predicts correctly the absolute configurations of several I derivs. but not that of I itself.

IT 77415-69-1P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation and diazaspirononane derivative from)

RN 77415-69-1 CAPLUS

CN Pentanedioic acid, 3,3-dicyano-, diethyl ester (9CI) (CA INDEX NAME)



L4 ANSWER 60 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1978:50444 CAPLUS

DOCUMENT NUMBER: 88:50444

ORIGINAL REFERENCE NO.: 88:7949a,7952a

TITLE: The chemistry of 2-oxopropanedinitrile (carbonyl cyanide); XIX. The ene synthesis using 2-oxopropanedinitrile and 1,3-dicarbonyl compounds

AUTHOR(S): Kociulek, K.; Leplawy, M. T.

CORPORATE SOURCE: Inst. Org. Chem., Tech. Univ. Lodz, Lodz, Pol.

SOURCE: Synthesis (1977), (11), 778-80

CODEN: SYNIBF; ISSN: 0039-7881

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 88:50444

AB Reaction of CO(CN)<sub>2</sub> with RCOCH<sub>2</sub>COR<sub>1</sub> (I; R = R<sub>1</sub> = Ph, 2,4,6-Cl<sub>3</sub>C<sub>6</sub>H<sub>2</sub>, Me; R = Me, F<sub>3</sub>C, R<sub>1</sub> = Ph) in ether at 0° was complete in 1 h and gave RCOCH(COR<sub>1</sub>)C(CN)<sub>2</sub>OH (II; R and R<sub>1</sub> as before) in 100% yield. Reaction of CO(CN)<sub>2</sub> with I (R = R<sub>1</sub> = OEt) at room temperature required 20 days and gave II in 43-66% yield.

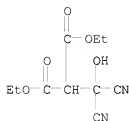
IT 65305-78-4P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation and reaction of, with aniline)

RN 65305-78-4 CAPLUS

CN Propanedioic acid, (dicyanohydroxymethyl)-, diethyl ester (9CI) (CA INDEX NAME)



L4 ANSWER 61 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1973:545978 CAPLUS

DOCUMENT NUMBER: 79:145978

ORIGINAL REFERENCE NO.: 79:23661a,23664a

TITLE: O,O-Dialkylthiophosphoric acid pseudochalcogen acyls

INVENTOR(S): Koehler, Helmut; Gerats, Irmtraut; Eichler, Gerhard; Kochmann, Werner

SOURCE: Ger. (East), 14 pp.

CODEN: GEXXA8

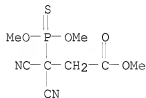
DOCUMENT TYPE: Patent

LANGUAGE: German

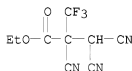
FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DD 95374	A1	19730212	DD 1971-156303	19710705 <--
PRIORITY APPLN. INFO.:			DD 1971-156303	A1 19710705
AB (MeO)2P(S)N(CN)CH2CO2R (I) and/or (MeO)2P(:NCN)SCH2CO2R (II) (R = Me or Et), prepared by reacting (MeO)2P(S)NNaCN with XCH2CO2R (X = Br or Cl), gave 95.0, 52.5 and 69.0% mortality for R = Me and 92.5, 51.0 and 55.0% for R = Et at 0.01, 1.0 and 0.05 weight % concentration, resp., against <i>Musca domestica</i> , <i>Sitophilus granarius</i> and <i>Tetranychus urticae</i> , resp. Analogs of I and II wherein the CO2R group was replaced by CONH2 and CONHMe, and (MeO)2P(S)C(CN)2CH2COR and (MeO)2P[:C(CN)2]SCH2COR (R = NHMe or OMe) were also prepared				
IT 50605-40-8P				
RL: SPN (Synthetic preparation); PREP (Preparation) (preparation of)				
RN 50605-40-8	CAPLUS			
CN Propanoic acid, 3,3-dicyano-3-(dimethoxyphosphinothioyl)-, methyl ester (CA INDEX NAME)				



L4 ANSWER 62 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1973:545936 CAPLUS  
 DOCUMENT NUMBER: 79:145936  
 ORIGINAL REFERENCE NO.: 79:23657a,23660a  
 TITLE: Reaction of some fluoroolefins with sodium cyanide  
 AUTHOR(S): Dyatkin, B. L.; Sterlin, S. R.; Zhuravkova, L. G.;  
 Martynov, B. I.; Knunyants, I. L.  
 CORPORATE SOURCE: Inst. Elementoorg. Soedin., Moscow, USSR  
 SOURCE: Zhurnal Organicheskoi Khimii (1973), 9(9),  
 1786-90  
 CODEN: ZORKAE; ISSN: 0514-7492  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Russian  
 GI For diagram(s), see printed CA Issue.  
 AB (CF<sub>3</sub>)<sub>2</sub>C:CF<sub>2</sub> reacted with 1 equiv of NaCN at -5 to 0° in dioxane  
 containing H<sub>2</sub>O to give 38% (CF<sub>3</sub>)<sub>2</sub>C:CF(CN) (I), and with excess NaCN in THF  
 containing H<sub>2</sub>O to give 55% NCC(CF<sub>3</sub>)<sub>2</sub>CH(CN)<sub>2</sub>, a C-H acid of pK<sub>a</sub> 2.12; analogous  
 treatment of (CF<sub>3</sub>)<sub>2</sub>C:CFPh and (CF<sub>3</sub>)<sub>2</sub>CHCO<sub>2</sub>Et yielded 49% NCC(CF<sub>3</sub>)<sub>2</sub>CHPhCN  
 and 51% EtO<sub>2</sub>CC(CN)(CF<sub>3</sub>)CH(CN)<sub>2</sub>, resp., after neutralization. Under  
 similar conditions, CF<sub>3</sub>CF:CF<sub>2</sub> afforded 59% CF<sub>3</sub>[C(CN)<sub>2</sub>]<sub>2</sub>Na, although its  
 acid could not be isolated, and (CF<sub>3</sub>)<sub>2</sub>C:CFOEt gave 3% (CF<sub>3</sub>)<sub>2</sub>C:C(CN)OEt. I  
 reacted with H<sub>2</sub>SO<sub>4</sub> and EtOH to give 25% (CF<sub>3</sub>)<sub>2</sub>C:CFCO<sub>2</sub>Et, with HCl in EtOH  
 to give 20% HOC(CF<sub>3</sub>)<sub>2</sub>CHFCONH<sub>2</sub>, with Et<sub>2</sub>NH to give 43% (CF<sub>3</sub>)<sub>2</sub>C:C(CN)N<sub>2</sub>,  
 with PhNH<sub>2</sub> to give 60% (CF<sub>3</sub>)<sub>2</sub>CHC(CN):NPh, and with concentrated H<sub>2</sub>SO<sub>4</sub> to give  
 84% iminolactone (II; R = H), which was converted to its Hg salt (II; R =  
 1/2 Hg) with HgO in refluxing aqueous Me<sub>2</sub>CO.  
 IT 50616-04-1P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)  
 RN 50616-04-1 CAPLUS  
 CN Propanoic acid, 2-cyano-2-(dicyanomethyl)-3,3,3-trifluoro-, ethyl ester  
 (CA INDEX NAME)



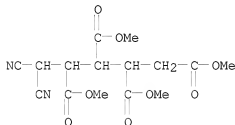
L4 ANSWER 63 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1965:462918 CAPLUS  
 DOCUMENT NUMBER: 63:62918  
 ORIGINAL REFERENCE NO.: 63:11492h  
 TITLE: Reaction of acetylenic esters with cyanoacetic ester  
 and pyridine  
 AUTHOR(S): Bamfield, P.; Crabtree, A.; Johnson, A. W.  
 CORPORATE SOURCE: Univ. Nottingham, UK  
 SOURCE: Journal of the Chemical Society (1965)  
 4355-62  
 CODEN: JCSOA9; ISSN: 0368-1769  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 AB Modified structures are suggested for the yellow and the blue adducts from

dimethyl acetylenedicarboxylate, Et cyanoacetate, and pyridine, which were originally prepared and formulated by Diels. The reaction of Me phenylpropiolate, Et cyanoacetate, and pyridine leads to a 1:1:1-adduct in which the pyridine has suffered ring-fission. Various reactions of the adducts are discussed.

IT 1289-25-4  
(Derived from data in the 7th Collective Formula Index (1962-1966))  
RN 1289-25-4 CAPLUS  
CN 1,2,3,4-Pentametetracarboxylic acid, 5,5-dicyano-, tetramethyl ester, compd. with pyridine (1:1) (8CI) (CA INDEX NAME)

CM 1

CRN 45287-28-3  
CMF C15 H18 N2 O8



CM 2

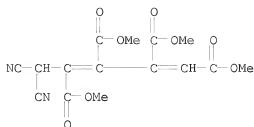
CRN 110-86-1  
CMF C5 H5 N



IT 100150-98-9P, Pentadiene-1,2,3,4-tetracarboxylic acid, 5,5-dicyano-, tetramethyl ester, compound with pyridine (1:1)  
RL: PREP (Preparation)  
(preparation of)  
RN 100150-98-9 CAPLUS  
CN Pentadiene-1,2,3,4-tetracarboxylic acid, 5,5-dicyano-, tetramethyl ester, compd, with pyridine (7CI) (CA INDEX NAME)

CM 1

CRN 100150-97-8  
CMF C15 H14 N2 O8



CM 2

CRN 110-86-1

CMF C5 H5 N



L4 ANSWER 64 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1964:484793 CAPLUS

DOCUMENT NUMBER: 61:84793

ORIGINAL REFERENCE NO.: 61:14826g-h,14827a-c

TITLE: 1-Halo-1,2,3,3-tetra(negatively substituted)propanes and their salts

INVENTOR(S): Martin, Elmore L.

PATENT ASSIGNEE(S): E. I. du Pont de Nemours &amp; Co.

SOURCE: 6 pp.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3133084		19640512	US	19600624 <--
PRIORITY APPLN. INFO.:			US	19600624

AB Compds. of the general formula  $[XC(Z):C(Y)C(A)R]-M^+(I)$ , where A, R, Y, Z are electron withdrawing groups such as CN, CO<sub>2</sub>Et, Bz, or SO<sub>2</sub>Ph, X is Cl or F, and M is H, Na, K, or a substituted ammonium ion, are dyes for natural and synthetic fibers. Thus, H<sub>2</sub>C(CN)<sub>2</sub> 79 in tetrahydrofuran (II) 220 was added with stirring to a dispersion 52 of 51.2% NaH in mineral oil and II 660 at 5-10° during 15 min., the mixture stirred 30 min., then dichlorofumaronitrile 44 in II 220 added during 15 min., II vacuum-distilled at 35-40°, the residual yellow solid dissolved in H<sub>2</sub>O 250, the pH adjusted to 8 with CO<sub>2</sub>, then Et<sub>4</sub>NBr 100 in H<sub>2</sub>O 200 parts added slowly with stirring, the mixture cooled to 5°, and the yellow crystals of I (A = R = Y = X = CN, Z = Cl, M = Et<sub>4</sub>N) (III) filtered, washed with 1% Et<sub>4</sub>NBr, and then H<sub>2</sub>O. The cake was dissolved in H<sub>2</sub>O 3500 at 100°, decolorizing carbon 10 added, the solution clarified, cooled to 5°, the long yellow needles filtered, washed with H<sub>2</sub>O and air-dried, giving 70

parts III, m. 129-31°,  $\lambda_{\text{maximum}}$  387 m $\mu$ ,  $\epsilon$  = 18,200 (MeOH) yellow on cellulose acetate and nylon, brownish yellow on wool and silk. Similarly, other I were prepared as tabulated below: X, Z, Y, A, R, M, % yield, m.p., color,  $\lambda$  (m $\mu$ ) maximum,  $\epsilon$ ; Cl, PhN(CO-)2, CN, CN, Me4N, 31 230-5° (decompose), orange, 468, 12,200; Cl, CO2Me, CO2Me, CN, CN, Et4N, 82, 88-90°, yellow, 335, 29,400; Cl, CN, CN, CO2Et, CO2Et, H, 100, b1, 115-20°, yellow (Na salt), -, -; Cl, Bz, Bz, CN, CN, Me4N, 39, 210-12° (decompose), yellow, 416, 27,000; F, CF3, CF3, CN, CN, Pr4N, 81, 84-6°, yellow, -, -; Cl, CN, CN, CN, CN, Me4N, -, 217-18° (decompose), yellow, 386, 17,600; Cl, CN, CN, CN, CN, Pr4N, -, 74-6° (decompose), yellow, 386, 18,100; Cl, CN, CN, CN, CN, Et3NH, -, 63-5° (decompose), yellow, 387, 17,200; Cl, CN, CN, CN, CO2Et, Et4N, 56, 70-2°, yellow, 400, 15,700; Cl, CN, CN, CN, SO2C6H4Me-4, Me4N, 73, 124-6° (decompose), yellow, 387, 17,000; Cl, CN, CN, CN, Bz, Me4N, -, 159-61°, yellow, 414, 17,100; Cl, CN, CN, CN, Bz, Et4N, 30 118-19°, yellow, 420, 16,200; Cl, CN, CN, CN, CN, Pr4N, -, 109-10°, yellow, 412, 17,600; Cl, CF3, CF3, CN, CN, Et4N, 64, 84-5°, yellow, -, -; F, -CF2CF2-, CN, CN, Na, -, -, orange, -, -; Cl, CN, CN, Bz, Bz, Me4N, -, 167°9°, yellow, 422, 8000; Cl, CN, CN, CN, CONHPh, K, -, -, red, -, -; Cl, CN, CN, SO2Ph, SO2Ph, Me4N, -, -, yellow, -, -; Cl, CN, CN, Bz, CO2Et, H, 20, 97-8°, colorless, -, -; Cl, CN, CN, Bz, CO2Et, Na, yellow;

IT 98469-37-5P, Ammonium, tetraethyl, 1,2-dicarboxy-1-chloro-3,3-dicyanopropenide, dimethyl ester

RL: PREP (Preparation)

(preparation of)

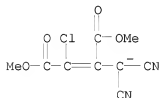
RN 98469-37-5 CAPLUS

CN Tetraethylammonium 1,2-dicarboxy-1-chloro-3,3-dicyanopropenide, dimethyl ester (7CI) (CA INDEX NAME)

CM 1

CRN 98469-36-4

CMF C9 H6 Cl N2 O4



CM 2

CRN 66-40-0

CMF C8 H20 N

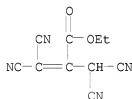


L4 ANSWER 65 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN  
 ACCESSION NUMBER: 1962:429396 CAPLUS  
 DOCUMENT NUMBER: 57:29396  
 ORIGINAL REFERENCE NO.: 57:5809h-i,5810c  
 TITLE: Nitration of cyclohexanecarboxylic acid to caprolactam  
 AUTHOR(S): Bigot, J. A.; Meijerink, Th. A. J.; Revallier, L. J.  
 CORPORATE SOURCE: Central Lab., Staatsmijnen, Geleen, Neth.  
 SOURCE: Recueil des Travaux Chimiques des Pays-Bas (1962), 81, 363-4  
 CODEN: RTCPA3; ISSN: 0165-0513  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 AB Cyclohexanecarboxylic acid (I) with nitryl hydropyrosulfate in oleum did not give the expected nitrocyclohexane, but 70% caprolactam (II) and a mixture of m-dinitrobenzene and nitrobenzene (total yield 22%, based on I). The mechanism of the reaction is unknown, but there is some evidence that removal of H<sub>2</sub>O from a nitro derivative is 1 of the steps involved. 1-Methyl-1-nitrocyclohexane with oleum gave a compound, C<sub>7</sub>H<sub>11</sub>NO (b2 79°, m. 48°), probably 1-methyl-1-nitrocyclohexene (or its rimer), a compound that could be isolated as such, since it could neither dehydrogenate to a C<sub>6</sub>H<sub>6</sub> derivative, nor disproportionate and subsequently rearrange to II.  
 IT 94211-18-4P, Acrylic acid, 3,3-dicyano-2-(dicyanomethyl)-, ethyl ester, compound with quinoline 94467-89-7P, Acrylic acid, 3,3-dicyano-2-(dicyanomethyl)-, ethyl ester, compound with NH<sub>3</sub>  
 RL: PREP (Preparation)  
 (preparation of)  
 RN 94211-18-4 CAPLUS  
 CN Acrylic acid, 3,3-dicyano-2-(dicyanomethyl)-, ethyl ester, compd. with quinoline (7CI) (CA INDEX NAME)

CM 1

CRN 94211-17-3

CMF C10 H6 N4 O2



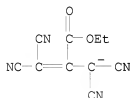
CM 2



CRN 91-22-5  
CMF C9 H7 N



RN 94467-89-7 CAPLUS  
CN 2-Propenoic acid, 3,3-dicyano-2-(dicyanomethyl)-, ethyl ester, ion(1-), ammonium (9CI) (CA INDEX NAME)



● NH<sub>4</sub><sup>+</sup>

L4 ANSWER 66 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1962:429395 CAPLUS

DOCUMENT NUMBER: 57:29395

ORIGINAL REFERENCE NO.: 57:5809e-h

TITLE: Base-catalyzed ring opening of diethyl  
1,1,2,2-tetracyanocyclopropane-3,3-dicarboxylate  
Regan, T. H.

AUTHOR(S): E. I. du Pont de Nemours & Co., Wilmington, DE

CORPORATE SOURCE: Journal of Organic Chemistry (1962), 27,  
2236-7

SOURCE: CODEN: JOCEAH; ISSN: 0022-3263

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB An example of a cyclopropane ring cleavage under very mild conditions was reported. CH<sub>2</sub>(CN)<sub>2</sub> (6.6 g.) in 17.4 g. di-Et oxomalonate was left 3 hrs. with one drop of base catalyst; the solid was collected and shown to be di-Et dihydroxymalonate. Fractionation of the yellow oil gave 11.7 g. di-Et 1,1-dicyanoethylene-2,2-dicarboxylate, b<sub>1</sub> 86°, n<sub>D</sub> 1.4628. An equimolar mixture of this compound and anthracene after heating at 150° gave crystalline product, m. 153.6-5.2° (alc.-H<sub>2</sub>O). The product resulting from 38 g. CH<sub>2</sub>(CN)<sub>2</sub> and 100 g. di-Et oxomalonate in 250 ml. alc. treated in the cold with 52 g. Br, the solution poured onto 1 kg. ice, and the oil crystallized when left overnight gave 71.5 g. di-Et 1,1,2,2-tetracyanocyclopropane-3,3-dicarboxylate (I), m. 129.6-31.2° (alc.-H<sub>2</sub>O). I (15 g.) was suspended in 500 ml. Et<sub>2</sub>O, treated with 15 g. dry NH<sub>3</sub>, stirred overnight and the mixture filtered to

give 11.4 g. solid, m. 192-201° (decomposition). The filtrate evaporated and the residue stirred with  $\text{CHCl}_3$  gave 0.5 g. yellow powder, m. 203° (decomposition). The  $\text{CHCl}_3$  solution evaporated gave Et carbamate, m. 46.6-8.6°. The yellow powder was ammonium 1,1,3,3-tetracyano-2-carbethoxypropenide (II). II in  $\text{H}_2\text{O}$  treated with a concentrated aqueous solution of quinolinium chloride gave quinolinium 1,1,3,3-tetracyano-2-carbethoxypropenide, m. 111.5-12.5°. Recrystn. from  $\text{H}_2\text{O}$  gave a hydrate, m. 51-2°.

IT 94211-18-4P, Quinoline, compound with Et 3,3-dicyano-2-(dicyanomethyl)-acrylate 94467-89-7P, Acrylic acid, 3,3-dicyano-2-(dicyanomethyl)-, ethyl ester, compound with  $\text{NH}_3$   
 RL: PREP (Preparation)  
 (preparation of)

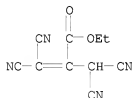
RN 94211-18-4 CAPLUS

CN Acrylic acid, 3,3-dicyano-2-(dicyanomethyl)-, ethyl ester, compd. with quinoline (7CI) (CA INDEX NAME)

CM 1

CRN 94211-17-3

CMF C10 H6 N4 O2



CM 2

CRN 91-22-5

CMF C9 H7 N



RN 94467-89-7 CAPLUS

CN 2-Propenoic acid, 3,3-dicyano-2-(dicyanomethyl)-, ethyl ester, ion(1-), ammonium (9CI) (CA INDEX NAME)



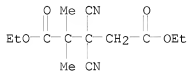
dimethyl-, diethyl ester

RL: PREP (Preparation)

(preparation of)

RN 858794-64-6 CAPLUS

CN Pentanedioic acid, 3,3-dicyano-2,2-dimethyl-, 1,5-diethyl ester (CA INDEX NAME)



L4 ANSWER 68 OF 68 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1938:911 CAPLUS

DOCUMENT NUMBER: 32:911

ORIGINAL REFERENCE NO.: 32:156d-i,157a-i,158a-e

TITLE: 2,3-Dioxopyrrolines, mononuclear substances related to isatin

AUTHOR(S): Mumm, Otto; Hornhardt, Hans

SOURCE: Berichte der Deutschen Chemischen Gesellschaft [Abteilung] B: Abhandlungen (1937), 70B, 1930-47

CODEN: BDCBAD; ISSN: 0365-9488

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB 5-Phenyl-2,3-dioxopyrroline (I) (C. A. 5, 703) is so extraordinarily similar in appearance and chemical properties to isatin that it may be considered as a mononuclear isatin, and it was hoped that by means of this very reactive substance light might be obtained on some of the controversial questions on isatin, especially the structure of its salts and derivs. The earlier work on I was accordingly resumed and attention was directed to the preparation of analogs of I containing an aliphatic residue instead of

Ph. In order not to weaken the ring unnecessarily, a residue (hexyl) of rather high mol. weight was chosen. As a tertiary residue might also favor the stability of the ring, Me<sub>3</sub>C was selected for a 2nd series of expts. It was intended to prepare the new compds. by the earlier method. Pinacolone and C<sub>6</sub>H<sub>13</sub>COME were condensed with HCO<sub>2</sub>R to the hydroxymethylene compds. which with NH<sub>2</sub>OH yielded the oxazoles through the intermediate oximes. The conversion of the oxazoles into the open-chain methylimide nitriles proceeded as expected and the formation from the nitriles of the desired pyrrolines with alc. HCl undoubtedly occurred, as evidenced by the appearance of the characteristic dark red color, but the products did not crystallize. The planned investigation was therefore continued with aromatic derivs., using p-tolyl instead of Ph compds. These tolyl compds., having higher m. ps., were considerably more stable and crystallized better. The starting point was α-p-tolylisoxazole (II). That the nitrile obtained from II was really p-tolylpyruvonitrile methylimide, RC(OH):CHC(:NMe)CN(R = p-MeC<sub>6</sub>H<sub>4</sub>) (III), was shown by the reaction with MeMgI, which gave the normal product, RC(OH)MeCH<sub>2</sub>C(:NMe)C(:NMgI) (IV), and also the compound RC(NH<sub>2</sub>)MeCH<sub>2</sub>C(:NMe)C(:NMgI)Me(V) when the Grignard compound was decomposed with NH<sub>4</sub>Cl instead of water. RC(OH)MeCH<sub>2</sub>C(:NMe)C(OH)(NHMgI)M

e (VI) was also formed by addition of H<sub>2</sub>O to IV under the influence of glacial AcOH. III merely treated in the cold with HCl in absolute alc. gave the blood-red di-HCl salt of 5-p-tolyl-2-oxo-3-methyliminopyrroline (VII). The previously assumed intermediate imido ester, RC(OH):CHC(:NMe)C(:NH)OEt (VIII), corresponding to the nitrile, was isolated as its white HCl salt, which readily changes, even in the absence of air, into the dark red derivative of VII. The distribution of the double bonds shown in III probably occurs only under the influence of the HCl, the free nitrile having the tautomeric structure RCOCH:C(NHMe)CN. The outstanding property of the dark red VII.2HCl is the ease with which the NMe group is replaced by O to form the brick-red 5-p-tolyl-2,3-dioxopyrroline (IX). As with isatin, NaOH cleaves the ring in IX to give  $\alpha$ -oxo- $\gamma$ -imino- $\gamma$ -p-tolylbutyric acid (X) through an intermediate intensely blue alkali salt. Attempts to liberate VII from its HCl salt were unsuccessful. Dilute aqueous alkali or NaHCO<sub>3</sub> gave, instead, the yellow-green pseudo base (XI), while excess of concentrated KOH yielded a dark red K salt, C<sub>12</sub>H<sub>11</sub>ON<sub>2</sub>K.2H<sub>2</sub>O, which regenerated XI with water. NH<sub>3</sub> in alc. replaces both the NMe group and the carbonyl O by NH and at the same time 1 mol. alc. is taken up with formation of a product, RC:CH.C(NH<sub>2</sub>)(OEt).C(:NH).NH (XII), similar in structure to XI; the dark red color immediately produced by HCl shows the ring has not been cleaved. PhNH<sub>2</sub> in alc. yields brick-red needles of the 3-phenylimino analog (XIII) of VII. With KOH and also with HCl, XIII forms salts which are red-violet in solution and almost black in the solid state. The HCl salt quant. splits off the HCl at high temps. in vacuo without changing to the brick-red of the free XIII, showing that the salt formation is accompanied by a simultaneous intramol. rearrangement. In water the HCl salt, like that of VII, is hydrolyzed to IX, but attempts to prepare the pseudo base were unsuccessful; instead was obtained XIII into which the K salt also changes on mere exposure to moist air. This difference in behavior and the very different colors show that the salts of VII and XIII have different structures. As with water and PhNH<sub>2</sub>, the dark red salt of VII also reacts with compds. having a reactive methylene group. Especially smooth, and under the mildest conditions, is the reaction with CH<sub>2</sub>(CN)<sub>2</sub> to give 5-p-tolyl-2-oxo-3-dicyanomethylenepyrroline (XIV), also obtained from IX or XIII. Surprisingly, XIV forms beautiful violet-black needles and dissolves, although difficultly, in alc. with red-violet color, whereas the corresponding isatin derivative is yellow-red, indicating a fundamental difference in structure. When the alc. solution of XIV is treated with a strong base, it immediately turns steel-blue, but, as with the salts of IX, the blue color quickly disappears and the ring is opened; acids precipitate the yellow cleavage product, RC(NH<sub>2</sub>):CHC[:C(CN)<sub>2</sub>]CO<sub>2</sub>H, m. 276°, probably in the form of the inner salt, which with boiling alc. HCl changes through the intermediate RC(NH<sub>2</sub>):CHC(CO<sub>2</sub>H):C(CO<sub>2</sub>H)C(:NH)OEt into the compound RC(NH<sub>2</sub>):CHC(CO<sub>2</sub>H):CHC(:NH)OEt (XV). X gently heated with dilute acids yields the dioxo acid. The ring in IX is also cleaved by piperidine, MeNH<sub>2</sub> and NH<sub>3</sub> to form compds. of the type RC(:NH)CH<sub>2</sub>COCONC<sub>5</sub>H<sub>11</sub> (XVI). PhNH<sub>2</sub> and CH<sub>2</sub>(CN)<sub>2</sub>, on the other hand, react with the 3-CO group, leaving the ring intact. The similarity of the dioxopyrrolines to isatin is also shown in their catalytic hydrogenation. There is first formed a light gray, alc.-insol. product (XVII) corresponding to isatide which in the air rapidly regenerates the original compound. If the hydrogenation is continued, the XVII redissolves, and cautious addition of water to the colorless alc. solution ppts. a completely air-stable crystalline product, RC(NH<sub>2</sub>):CHCH(OH)CO<sub>2</sub>H (XVIII). Reduction of XIII in alc. proceeds 1 step further, with addition of 2 mols. H and 1 mol. alc. to give the compound

RCH(NH<sub>2</sub>)CH<sub>2</sub>CH(NHPh)CO<sub>2</sub>Et (XIX). For the bearing of the above facts, especially the color phenomena, on the structures of the mononuclear isatins and their derivs., the original should be consulted.

Hydroxymethylenepinacolone dioxime (54% yield), m. 84°.

$\alpha$ -tert-Butylisoxazole, b<sub>760</sub> 156°; its methosulfate with KCN

in water at 0° gave 82% trimethylacetopyruvonnitrile methylimide, m.

42°, hydrolyzed by cold concentrated HCl to the pyruvic acid, crystals

with 1 H<sub>2</sub>O, m. 64°, and by dilute HCl to the amide, m. 115°;

in cold absolute alc. with HCl gas the nitrile imide gave a dark red oil which with 2 N NaOH or 50% AcOH yielded  $\alpha$ -oxo- $\alpha$ -imino- $\delta$ , $\delta$ -dimethylcaproic acid, m. 185° (gas evolution).

When the red oil was carefully freed from adhering HCl, simple solution in ordinary alc. resulted in ring cleavage (probably by the water in the

alc.), but AcOEt precipitated a crystalline substance, m. 186°, insol. in all solvents except alc. and water, which on gentle warming with water gave trimethylacetopyruvic acid methylimide, m. 183°.

Hydroxymethylenemethyl hexyl ketone oxime, m. 118°.

$\alpha$ -Hexylisoxazole, b<sub>11</sub> 97-8°, was analyzed as the chloroplatinate, C<sub>20</sub>H<sub>36</sub>O<sub>2</sub>N<sub>2</sub>PtCl<sub>6</sub>, obtained from the methosulfate with

PtCl<sub>4</sub>.  $\alpha$ , $\alpha$ -Dioxodecanonitrile  $\alpha$ -methylimide, oil

decomposing on distillation, even in a high vacuum;  $\alpha$ , $\alpha$ -dioxodecanamide, m. 99°. Hydroxymethylene-p-methylacetophenone

oxime (67% yield), m. 133°. II, m. 60°. III, light yellow,

m. 126°. IV (3 g. from 2 g. II and 2.2 mol. MeMgI boiled 2 h. in ether), yellow, m. 175° (decomposition); heated a short time or allowed

to stand 1 day at room temperature in glacial AcOH, it changed into VI, rhombic tables, red-brown in incident light, m. 183° (decomposition). V, m.

197°, soluble in AcOH with wine-red color, easily soluble in dilute HCl and repptd. by NaOH. VII.2HCl (78%), sinters and carbonizes at 183°;

picrate, intensely red, m. 192°. VIII.HCl, from III in cold dioxane with 0.662 N HCl in absolute alc., decomp. 145°. If in the

treatment of III with alc.-HCl water is present even only in traces the reaction proceeds in part in an entirely different way, giving in addition to

the dark red salt Me p-tolulypyruvate, m. 84°; free acid, crystals with 1 H<sub>2</sub>O, m. 143°. Ag salt of VII, red needles with 1 MeOH,

decomposing 172°. Cu salt, (C<sub>12</sub>H<sub>11</sub>ON<sub>2</sub>)<sub>2</sub>Cu.4H<sub>2</sub>O, green, m. 191° (decomposition). XII, m. 153°. IX, precipitated quant. in about 6 h. from

VII.2HCl in 20 parts cold water, m. 229-30°; a cold alc. suspension treated with somewhat less than 1 mol. EtOK-solution at once becomes

blue-violet and soon deposits the K salt, C<sub>11</sub>H<sub>8</sub>O<sub>2</sub>NK.2H<sub>2</sub>O, which is not very stable even when dry; one sample had become yellowish after 14 days.

Alc. IX treated with aqueous NaOH also immediately turns blue-violet but the color rapidly disappears and on cautious acidification X, m. 155°,

seps. Piperidine (XVI), m. 184°. Amide, C<sub>11</sub>H<sub>12</sub>O<sub>2</sub>N<sub>2</sub>.0.5H<sub>2</sub>O, m. 179°. Methylamide (0.5H<sub>2</sub>O), m. 169°. XVIII, turns brown

and carbonizes 245-50°. XIII, m. 237°. XIX, m.

123°. XIV was obtained in 92% yield; its melting or decomposition point is so extraordinarily high that it could not be determined XV.2HCl,

lemon-yellow, m. 148-9°.

IT 855234-21-8P, 3-Butenoic acid, 4-amino-2-(dicyanomethylene)-4-p-tolyl-

RL: PREP (Preparation)

(preparation of)

RN 855234-21-8 CAPLUS

CN 3-Butenoic acid, 4-amino-2-(dicyanomethyl)-4-(4-methylphenyl)- (CA INDEX NAME)

10/923,271

